

# **A NON-INVASIVE TECHNIQUE FOR COMPOSITIONAL ANALYSIS OF COAL**

A thesis submitted in partial fulfillment of the requirements for  
the award of the degree of

**M.Tech**

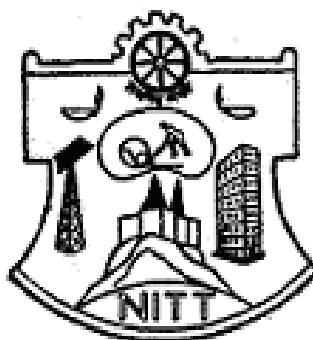
**in**

**Non-Destructive Testing**

**By**

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## **BONAFIDE CERTIFICATE**

This is to certify that the project titled **A NON-INVASIVE TECHNIQUE FOR COMPOSITIONAL ANALYSIS OF COAL** is a bonafide record of the work done by

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## ABSTRACT

India has some of the largest coal reserves in the world (approx. 267 billion tones). The increasing demand and use of energy internationally, especially in India is heavily dependent on coal. Indian coal industries mainly deals with bituminous and sub-bituminous coal in which the ash content varies from 25 to 75%. Quality of Coal (ash content in the coal) is one of the important factors which directly affects the power generation in the thermal power plant and the efficiency of blast furnace in steel plants. High ash content reduces the thermal efficiency of the coal. Moreover the ash content has to be controlled based on the plant's capacity and ash handling systems. The proximate analysis methods which are being employed now are less accurate and time consuming. The present work describes an Infrared (IR) Thermography based fast and low cost technique for the compositional analysis of coal taking into account the change in average temperature due to the variation in thermal absorptivity of coal constituents. Different coal samples with ash content varying in the range of 25.0 to 85.0 wt.% and moisture content from 1.0 to 6.0 wt.% have been collected from Coal Characterization Centre in CSIR-NML. Fixed quantities of these samples were uniformly heated using a microwave oven, for a duration of 40s which is just sufficient to create a difference between the sample constituents in the extent of their respective infrared emissions. The thermal image of the heated samples is captured by IR Thermography and the average temperature of these samples is evaluated from the thermal image. This experiment has been done with samples obtained from two different mines; Sambaleswari and Talcher mines. The composition of the coal samples in terms of their ash and moisture content is obtained from proximate analysis results. Calibration curves relating average temperature and the ash and moisture content in coal samples is obtained. Finally, a software program has been developed in LabVIEW for estimating the ash and moisture content in a fixed quantity of coal sample from the calibration curves. This technique can be used for the determination of ash and moisture content in coal samples without carrying out proximate analysis and thereby would be useful in finding the suitability of specific coals for individual combustion systems.

**Keywords:** IR Thermography, coal, ash content, moisture content, compositional analysis

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# CHAPTER 1

## INTRODUCTION

### 1.1 BACKGROUND

The increasing demand and use of energy internationally, especially in India is heavily dependent on coal. India has some of the largest coal reserves in the world (approx. 267 billion tones). The energy derived from coal in India is about twice that of energy derived from oil, whereas worldwide, energy derived from coal is about 30% less than energy derived from oil. Indian coal is of mostly sub-bituminous rank, followed by bituminous and lignite (brown coal). The mineral deposits of India are shown in the Fig. 1.1.



Fig. 1.1 Mineral deposits of India

Coal is the world's most abundant and widely distributed fossil fuel. It is a global industry that makes a significant economic contribution to the global economy. Coal is mined commercially in more than 50 countries and used in more than 70. Annual world coal consumption is about 5,800 million tons, of which about 75% is used for electricity production. This consumption is projected to nearly double by the year 2030 to meet the challenge of sustainable development and a growing demand for energy. Coal produces 40% of the world's electricity, which is double the share of its nearest competitors (gas and hydro) and coal is an essential element in over 65% of the world's steel production[1].

The International Energy Agency (IEA) predicts that world energy demand will grow around 60% over the next 30 years, most of it in developing countries. China and India are very large countries in terms of both population and land mass, and both have substantial quantities of coal reserves [2]. Together, they account for 70% of the projected increase in world coal consumption. Strong economic growth is projected for both countries (averaging 6% per year in China and 5.4% per year in India from 2003 to 2030), and much of the increase in their demand for energy, particularly in the industrial and electricity sectors, is expected to be met by coal.

Coal is a complex combination of organic matter and inorganic mineral matter formed over year from successive layers of fallen vegetation [3]. Coals are classified by rank according to their progressive alteration in the natural metamorphosis from lignite to anthracite. Coal rank depends on the volatile matter, fixed carbon, inherent moisture, and oxygen; although no single parameter defines a rank. Typically, coal rank increases as the amount of fixed carbon increases and the amount of ash and moisture decreases.

Chemical composition of the coal is defined in terms of its proximate and ultimate (elemental) analysis. The parameters of proximate analysis are moisture, volatile matter, ash, and fixed carbon. Elemental or Ultimate analysis encompasses the quantitative determination of carbon, hydrogen, nitrogen, sulfur, and oxygen. The calorific value  $Q$ , of coal is the heat liberated by its complete combustion with oxygen.  $Q$  is a complex function of the elemental composition of the coal. Gross

Calorific value Q is mostly determined by experimental measurements. A close estimate can be made with the Dulong formula:

$$Q = (144.4 \%[C]) + (610.2 \%[H]) - (65.9 \%[O]) + (0.39 \%[O]^2)$$

Q is given in Kcal/kg or Btu/lb.

Indian coal is classified by grades defined on the basis of Useful Heat Value (UHV). UHV is an expression derived from ash and moisture contents for coals as per the Government of India notification. The ultimate analysis of coal used in power plants in India is readily not available. Ultimate analysis of D, E, and F grade coal is provided by Central Fuel Research Laboratory (CFRI), Jharkhand, India. Ultimate analysis of coal used at Dadri, Rihand, Singrauli, Chandrapur, and Dahanu power plants is obtained from National Energy Technology Laboratory, Pittsburgh, USA. The Neyveli Lignite Corporation provided by personal communication, the ultimate analysis for lignite is used at the Neyveli and Kutch power plants. These are presented in Tables 1.1 and 1.2.

**Table 1.1 Elemental analysis, moisture content, and grades of typical Indian coals**

Coal Grade	C(%)	H(%)	S(%)	N <sub>2</sub> (%)	O <sub>2</sub> (%)	A(%)	M(%)	NCV (Kcal/Kg)	UHV (cal/gm)
D	33.1	2.46	0.44	0.83	NA	25.9	7.2	4999.0	4332.0
D	30	2.48	0.57	0.69	NA	27.1	2.9	5555.0	4760.0
D	32.3 1	2.12	0.4	0.78	NA	25	7.3	5068.0	4442.0
E	37.9	2.4	0.53	0.8	6%	30.4	7.5	4529.0	3670.0
F1	41.8 7	3.33	0.56	0.94	6%	34.0 7	7.8	4137.0	3122.0
F2	44.4 7	3.37	0.35	0.99	6%	36.3	8.4	3833.0	2731.0
Average of E and F2	41.1 9	2.89	0.44	0.9	0.06	33.3 5	7.95	4182.0	-

**Table 1.2 Elemental analysis and moisture content of the coal used at the seven power plants in India**

Coal	C(%)	H(%)	S(%)	N <sub>2</sub> (%)	O <sub>2</sub> (%)	A(%)	M(%)	NCV
------	------	------	------	--------------------	--------------------	------	------	-----

Dadri	40.3	4.16	0.5	0.9	15.92	38.22	NA	NA
Rihand	37.74	3.26	0.39	0.73	14.65	43.23	NA	NA
Singrauli	50.22	4.78	0.33	1.09	17.25	26.33	NA	NA
Chandrapur	37.69	2.66	0.8	1.07	5.78	47.0	5	3649.9
Dahanu	42.39.0	3.73	0.39	0.82	14.21	38.46	5.93	3986.37
Neyveli Lignite	26.09	2.33	1.5	0.24	16.33	7.0	47	2229.0
Kutch Lignite	28.33	3.03	2.25	0.88	13.94	15.0	36	2900.0

NA: Not Available

C: Carbon

H: Hydrogen

S: Sulfur

N: Nitrogen

CV: Calorific value

A: Ash

M: Moisture

UHV: Useful heat value =  $8900 - 138(A+M)$

GCV: Gross Calorific Value =  $(UHV + 3645 - 75.4 M)/1.466$

NCV: Net Calorific Value =  $GCV - 10.02M$

Relationship of GCV, UHV, and NCV is empirical.

Ultimate analysis of typical United States coals is given in Table 1.3 for the sake of comparison.

**Table 1.3 Ultimate analysis of typical US coals**

		<b>Chandrapur (India)</b>	<b>Ohio (USA)</b>
1	Fixed carbon	27.5%	44.0
2	Total carbon	37.69%	64.2%
3	Hydrogen	2.66%	5.0 %
4	Nitrogen	1.07%	1.3 %
5	Oxygen (difference)	5.78%	11.8 %
6	Sulfur	0.8%	1.8 %
7	Ash	47%	16%

8	Total moisture	5%	2.8
9	Gross calorific value Kcal/Kg	3400	6378
10	Coal per unit of electricity (Kg/KWH)	0.77	0.36

The coal properties including calorific values differ depending upon the colliery. The calorific value of the Indian coal (~15 MJ/kg) is less than the normal range of 21 to 33 MJ/Kg (gross).

Coal as a fossil fuel is used to supply heat energy in many power plants, boilers and furnaces in various industries all over the world. Coal ash is the oxidized residue left after burning coal and is an important factor in determining the calorific value of coal. Coal ash consists mainly of alumina, silica and iron oxide with small quantities of many other metal oxides.

Even as demand grows, society expects cleaner energy with less pollution and an increasing emphasis on environmental sustainability. The coal industry recognizes it must meet the challenge of environmental sustainability; in particular it must reduce its greenhouse gas emissions if it is to remain a part of a sustainable energy future.

The ash content in Indian coal ranges from 25% to 75%. In power plants, when the ash content increases from 6% to 75%, the adverse effects on the system are as follows[5]:

- Total boiler area requirement increases by 69%.
- Efficiency of boiler fans drop by 22%–27%.
- Induced draft (ID), forced draft (FD), and primary air (PA) fans show an increase in specific energy consumption (SEC) of around 30%, 6%–14%, and 2%–7% respectively.
- Drum mills show an increase in SEC of 115% while ball-race mills and bowl mills show an increase in SEC of 30%.
- The gross and net overall efficiencies are reduced to 77% and 66% of their original values.
- The ratio of the specific fuel consumption at a given ash content to that at standard ash of 6% increases from 1 to 10.
- Specific fuel consumption (SFC) gross increases from 0.35 to 3.0.

Determination of ash and moisture present in coals, by chemical analysis method is time consuming and one cannot have an on-line analysis. This absence of real-time online analysis data creates problems for plant operators and quality assurance managers. Hence an alternate technique is needed to study the quality of coals in terms of ash and moisture present in them and henceforth find the suitability of specific coals for individual combustion systems.

## **1.2 OBJECTIVE**

The major objectives of this project are:-

- To provide a faster way to determine the ash and moisture content in coal and thereby to find the suitability of specific coals for individual combustion systems.
- To develop a software for obtaining the calibration curve and henceforth predict ash and moisture in the coal samples.

## **1.3 PLAN OF WORK**

- Collection of coal samples from different mines.
- Optimization of heating source and heating time for IR thermography of coal samples.
- IR image capturing and analysis.
- Chemical analysis of coal.
- Development of Calibration Curve for determination of ash and moisture content in coal.
- Software development for prediction of ash and moisture content in coal.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 LIMITATIONS OF AVAILABLE TECHNIQUES**

Standard test methods for routine coal analysis include those mentioned in American Society for Testing and Materials (ASTM) and, with limited application, those of the International Organization of Standardization (ISO)[6]. In analyzing coal research samples for their chemical composition, it is apparent that certain current standard test methods require modification or they are not applicable. Coal research, and possibly most new uses of coal, requires higher standards of quality control and accuracy than that are currently quoted in many of the existing standard test methods. These factors become increasingly more important when economic decisions have to be made based on the validity, i.e., accuracy, of coal analysis data. The problems related with ash and moisture analysis are given below.

##### **2.1.1 Limitations of Moisture Analysis Techniques**

Determining the moisture in the analysis by weight loss at 104-110°C presents several problems, especially if the current ASTM method D3173 (1) is used. The ASTM method is unsuitable for low-rank coals because it does not use an inert gas as the purge gas and recommends too short a drying period. Due to oxidation gain in weight as the sample reacts with oxygen in the air can take place, and not all of the 104-110°C moisture is removed during the recommended one-hour drying time. In addition, with certain low-rank coals, decarboxylation during the drying period can result in weight loss.

##### **2.1.2 Limitations of Ash Analysis Techniques**

The ash in coal is the non-combustible residue that remains when coal is burned. In the ASTM method D3174 (3), the coal sample is placed in a cold furnace and heated gradually so the temperature reaches 450 to 500°C in one hour and 700 to 750°C at the end of the second hour. The ISO method 1171 (4) recommends 815°C final temperature. In both methods the sample is ignited at the appropriate final temperature to constant weight. Furthermore, there is difference of opinion as to the proper rate of



heating for the determination of ash in coals high in calcite and pyrite[7]. In the determination of ash, particularly in coals containing appreciable amounts of pyrite and calcite, there is danger that the ash will retain indefinite amounts of sulfur which causes erratic results. Although it is widely recognized that the rate of heating influences the sulfur retention, there is disagreement as to how fast the heating rate may be and still give reliable test results.

Therefore, a relatively capital inexpensive, reliable online sensing system, which is also faster compared to the methods currently in use, is desirable for the compositional analysis of coals.

## 2.2 COAL CLASSIFICATION AND ANALYSIS

Coal is classified into three major types namely anthracite, bituminous, and lignite. However there is no clear demarcation between them and coal is also further classified as semi- anthracite, semi-bituminous, and sub-bituminous[8]. Anthracite is the oldest coal from geological perspective. It is a hard coal composed mainly of carbon with little volatile content and practically no moisture. Lignite is the youngest coal from geological perspective. It is a soft coal composed mainly of volatile matter and moisture content with low fixed carbon. Fixed carbon refers to carbon in its free state, not combined with other elements. Volatile matter refers to those combustible constituents of coal that vaporize when coal is heated. The common coals used in Indian industry are bituminous and sub-bituminous coal. The gradation of Indian coal based on its calorific value is given in the table 2.1

**Table 2.1 Gradation of Indian coal**

Grade	Calorific Value Range ( in kCal/kg)
A	Exceeding 6200
B	5600 – 6200
C	4940 – 5600
D	4200 – 4940
E	3360 – 4200
F	2400 – 3360
G	1300 – 2400

Normally D, E and F coal grades are available to Indian Industry.

### **2.2.1 Analysis of Coal**

Analysis of coal is mostly done by two methods: ultimate analysis and proximate analysis[9]. The ultimate analysis is used to determine all coal constituent elements, solid or gaseous while the proximate analysis technique determines the fixed carbon, volatile matter, moisture and ash percentages. The ultimate analysis is carried out in a properly equipped laboratory by a skilled chemist, while proximate analysis can be done with a simple apparatus. It may be noted that proximate has no connection with the word “approximate”.

### **2.2.2 Proximate Analysis**

Proximate analysis indicates the percentage by weight of the Fixed Carbon, Volatiles, Ash, and Moisture Content in coal[10]. The amounts of fixed carbon and volatile combustible matter directly contribute to the heating value of coal. Fixed carbon acts as a main heat generator during burning. High volatile matter content indicates easy ignition of fuel. The ash content is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling systems of a furnace.

#### **(i) Measurement of Moisture**

Determination of moisture is carried out by placing a sample of powdered raw coal of size 200-micron size in an uncovered crucible and it is placed in the oven kept at  $108 \pm 2$  °C along with the lid. Then the sample is cooled to room temperature and weighed again. The loss in weight represents moisture.

#### **(ii) Measurement of Volatile Matter**

Fresh sample of crushed coal is weighed, placed in a covered crucible, and heated in a furnace at  $900 \pm 15$  °C. The sample is cooled and weighed. Loss of weight represents moisture and volatile matter. The remainder is coke (fixed carbon and ash).

#### **(iii) Measurement of Carbon and Ash**

The cover from the crucible used in the last test is removed and the crucible is heated over the Bunsen burner until all the carbon is burned. The residue is weighed, which is the incombustible ash. The difference in weight from the previous weighing is the fixed carbon. In actual practice Fixed Carbon or FC is derived by subtracting from 100 the value of moisture, volatile matter and ash.

A typical proximate analysis of various grades of coal is given in the Table 2.2.

<b>TABLE 2.2 TYPICAL PROXIMATE ANALYSIS OF VARIOUS COALS (IN PERCENTAGE)</b>			
<b>Parameter</b>	<b>Indian Coal</b>	<b>Indonesian Coal</b>	<b>South African Coal</b>
Moisture	5.98	9.43	8.5
Ash	38.63	13.99	17
Volatile matter	20.70	29.79	23.28
Fixed Carbon	34.69	46.79	51.22

### **2.2.3 Significance of Various Parameters in Proximate Analysis**

#### **(a) Fixed carbon:**

Fixed carbon is the solid fuel left in the furnace after volatile matter is distilled off. It consists mostly of carbon but also contains some hydrogen, oxygen, sulphur and nitrogen not driven off with the gases. Fixed carbon gives a rough estimate of heating value of coal.

#### **(b) Volatile Matter:**

Volatile matters include methane, hydrocarbons, hydrogen and carbon monoxide, and incombustible gases like carbon dioxide and nitrogen found in coal. Thus the volatile matter is an index of the gaseous fuels present. Typical range of volatile matter is 20 to 35%.

Volatile Matter

- Proportionately increases flame length, and helps in easier ignition of coal.
- Sets minimum limit on the furnace height and volume.
- Influences secondary air requirement and distribution aspects.
- Influences secondary oil support

#### **(c) Ash Content:**

Ash is an impurity that will not burn. Typical range is 5 to 40%.

Ash

- Reduces handling and burning capacity of coal
- Increases handling costs.
- Affects combustion efficiency and boiler efficiency
- Causes clinkering and slagging.

#### **(d) Moisture Content:**

Moisture in coal must be transported, handled and stored. Since it replaces combustible matter, it decreases the heat content per kg of coal. Typical range is 0.5 to 10%

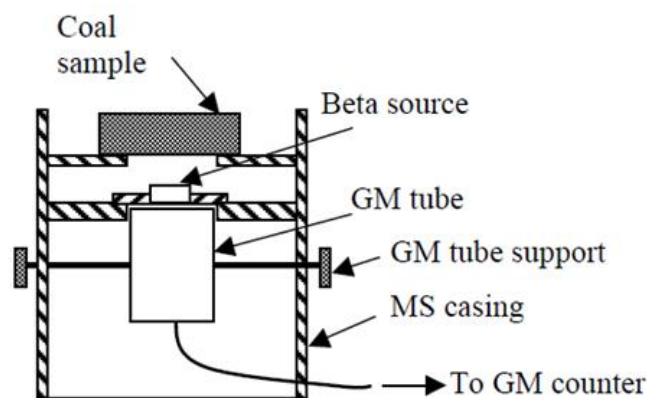
#### Moisture

- Increases heat loss, due to evaporation and superheating of vapour
- Helps, to a limit, in binding fines.
- Aids radiation heat transfer.

## 2.3 TECHNIQUES USED FOR DETERMINATION OF ASH IN COAL

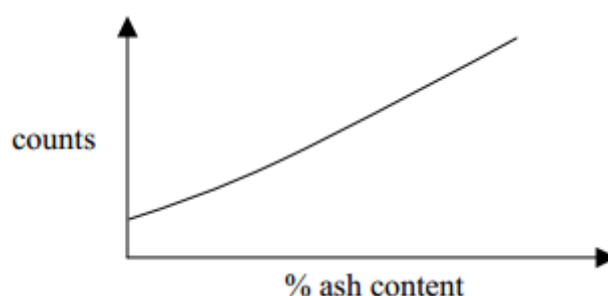
### 2.3.1 Measurement of ash content in coal using beta ray back scattering technique

Method of on-line measurement of ash content in coal by back scattering is based on the difference in atomic number ( $Z$ ) of coal and ash constituents. The atomic number of elements O, Al, Si, Fe of which ash is essentially composed, differ quite extensively from the atomic number of carbon and therefore those interactions of radiation with matter, which depend upon the atomic number can be utilized to determine the ash percentage in coal. The components of the coal (H, C, N, O) have lower atomic number than those of ash (Mg, Al, Si, S, Ca, Fe). This makes it a two component system. The intensity of the reflected beam with the same ash composition is a function of the ash content. The set up for beta backscattering for ash measurement in coal is shown in the Fig. 2.1.



**Fig. 2.1 Beta backscattering set up for ash measurement in coal**

For the ash analysis one would choose a high energy  $\beta$  source with a fairly large half life to minimize the error due to decay. Activity should be high enough to give required count in reasonable time limit. The ideal selection is Sr90-Y90. (half life = 28 years,  $E_{max} = 2.2$  Mev and of activity 4  $\mu$  curie.) GM tube is used as the detector with the necessary counting system. The graph between ash content and count rate is given in the Fig. 2.2. The operating voltage of the detector has to be within the plateau region. Various coal samples of known ash content are prepared. The thickness of the sample should be more than the saturation thickness to ensure that the back scattered intensity is independent of the thickness of the sample.



**Fig. 2.2 Plot of ash content v/s count rate**

### **2.3.2 Proximate Analysis of Coal and Coke using the STA 8000 Simultaneous Thermal Analyzer**

Proximate analysis has long been used to determine the rank of coals by separating volatile components, fixed carbon and inert components. Because of the wide ranging quality of coal products and the commercial value of ranking these products the need for good methods is obvious. To meet these needs there are ASTM tests to perform these separations separately using specialized industrial equipment. The STA8000 Simultaneous Thermal Analyzer (STA) is able to analyze coal and coke to obtain Proximate Analysis data of volatiles, fixed carbon and ash using 10 to 100 milligram samples[12].

The instrument requirements needed are the ability to accurately record the weight of a sample as it is heated over a temperature range and held isothermally at designated temperatures, then change the sample's environmental atmosphere from inert to oxidizing. The PerkinElmer STA 8000 which is shown in the Fig. 2.3 easily meets these requirements, including accurate temperature and gas control, a balance with microgram sensitivity, and software to facilitate and automate the analysis.

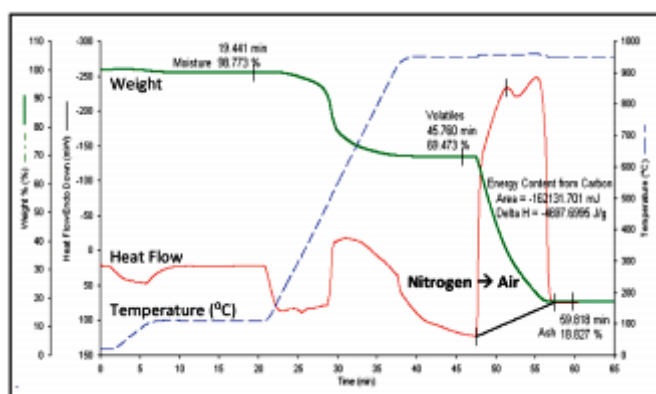


**Fig. 2.3 The STA 8000 Simultaneous Thermal Analyzer.**

The weighing mechanism is ideally suited for routine quality testing use. The STA 8000 uses a top loading balance. This makes it easier to load, and when loading there is no force applied to a long balance arm or hang-down wire that could result in damage to the balance system.

The sample is weighed and loaded at 25 °C, heating to 110 °C, holding for 10 minutes then heating to 950 °C where the temperature was held until constant weight was achieved in nitrogen. Then the sample atmosphere was switched to air (somewhat diluted by the balance purge). When the weight was again constant after the combustion of the fixed carbon component the analysis was ended and the apparatus cooled for the next analysis.

For determination of total volatiles the sample is heated from 107 °C to 950 °C and held isothermally to drive off all volatile components. After the switch to an oxidizing atmosphere combustion at 950 °C is rapid and complete and the weight loss gives the fixed carbon. After combustion, the remaining residue is the ash content of the coal, which can be read directly from the weight. The proximate analysis using STA 8000 is shown in the Fig. 2.4.

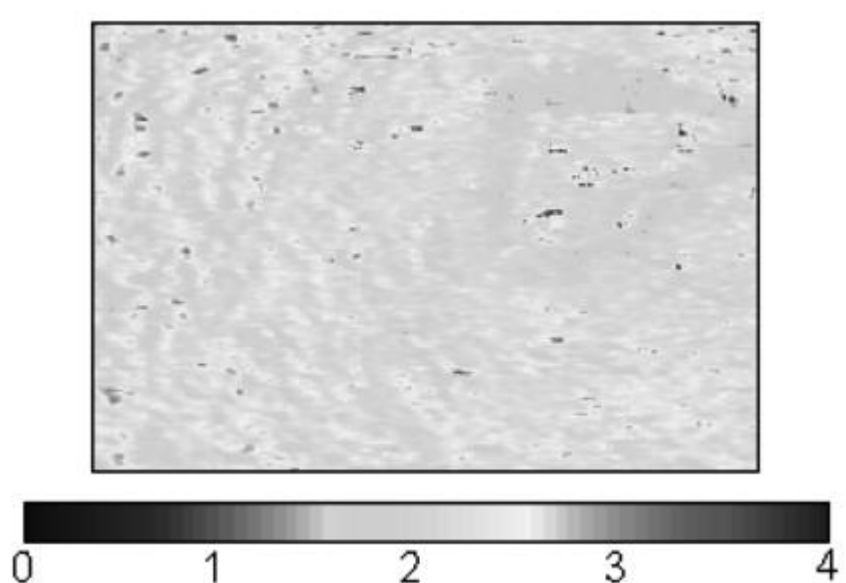


**Fig. 2.4 Proximate analysis of coal using the STA 8000.**

### 2.3.3 Real-time coal or ore grade sensor

A real-time coal content or ore grade sensor can be used during exploration, mining, and processing operations. The sensor is developed based on advanced multi-spectral and hyper-spectral imaging technology that is currently used in airborne and satellite applications.

This project utilizes the unique spectral characteristics of coal and platinum or palladium to quantify coal content and ore grade. Because the instrument will utilize imaging technology that requires only a few hundredths of a second per measurement, the mineral content across a working surface or in particulate matter will be rapidly measured. Using modern reflectance spectroscopy and digital image processing techniques, the spectral signatures of target minerals will be used to generate and easily interpret a false color map indicating mineral content in coal or ore. This will allow for greater selectivity as well as decrease environmental impacts and energy requirements in exploration, mining, and processing activities. The processed microspectral image of ore sample is shown in the Fig.2.5.



**Fig. 2.5 Processed microspectral image of ore sample**

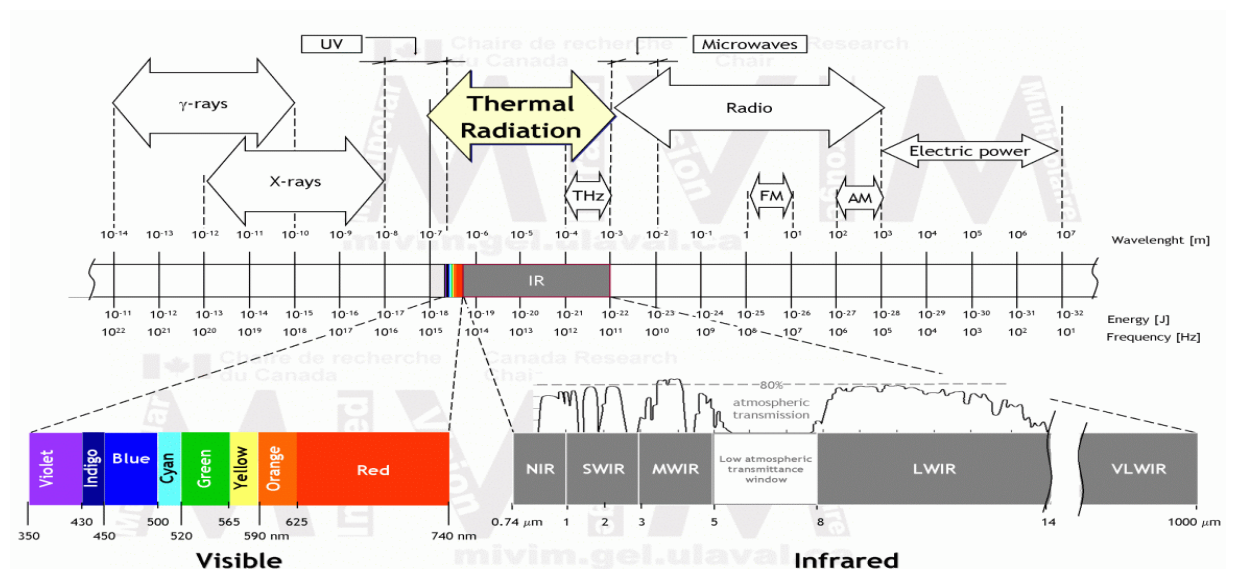
The dark areas above show false colouring used to highlight the presence of sulfides within the ore.

It is evident from the literature study that IR Thermography based techniques have not yet been explored for compositional analysis of coal. IR Thermography has been found to be a useful technique for identification and gradation of alumina-rich iron ores [15] and this technique may be further explored for compositional analysis of

coal. As this technique is based on the relative difference in thermal absorptivity of ore constituents for studying their quality, this can also be used to determine the ash and moisture content in coal. Therefore, the prime objective of this project is to develop an IR thermography based non-invasive technique for faster compositional analysis of coal.

## 2.4 INFRARED THERMOGRAPHY

All objects with surface temperature above absolute zero emit electromagnetic radiation that is uniquely related to object temperature. There is no fundamental difference between radiations in the different bands of the electromagnetic spectrum. They are all governed by same laws and the only difference is due to the variation in wavelength as shown in Fig. 2.6 of the electromagnetic spectrum.



**Fig. 2.6 Electromagnetic Spectrum.**

Infrared Thermography is the science of acquisition and analysis of thermal information by using non-contact thermal imaging devices. Infrared (IR) emission is



electromagnetic radiation with longer wavelengths than those of visible light but shorter than microwaves. The infrared band spans from 0.76  $\mu\text{m}$  to 1000  $\mu\text{m}$ , also called “thermal band”. The infrared spectrum is further divided into 3 regions:-

- [1] Short wave/Near Infrared (0.76 - 2  $\mu\text{m}$ )
- [2] Medium wave/Middle Infrared (2 - 4  $\mu\text{m}$ )
- [3] Long wave/Far Infrared (4 - 1000  $\mu\text{m}$ )

Thermography is one of the widely used non-destructive test (NDT) techniques. Some of the advantages of IR Thermography are:

- It is a non-contact method of temperature measurement which allows collecting data from hazardous or physically inaccessible objects and does not intrude upon or affect the target at all.
- It has fast inspection rate (up to few  $\text{m}^2$  at a time).
- It keeps the user safe since measurement is non-contact and no harmful radiations are involved.
- It also facilitates measurement of moving targets and enables capturing of fast changing thermal patterns in real time.
- There is no risk of contamination and no mechanical effect on the surface of the object; thus wear free. Lacquered surfaces are not scratched and soft surfaces can also be measured.
- The thermal imaging technique is being widely used in various fields which include: inspections for predictive maintenance, non-destructive evaluation of thermal and mechanical properties, building science, agriculture, military reconnaissance, weapons guidance and medical imaging.

As with all NDT methods, thermo graphic inspection also has its limitations, which include:

- Difficulty in obtaining a quick, uniform and highly energetic thermal stimulation over a large surface.
- Effects of thermal losses which induce spurious contrasts affecting the reliability of interpretation.
- It is a boundary technique which allows inspecting a limited thickness of material under the surface and detects defects which results in measurable change in thermal properties.
- The equipment cost is usually higher.

### 2.4.1 Governing Equations

The electromagnetic radiation depends upon two parameters; its wavelength  $\lambda$  and intensity  $E$  and these two parameters affect the surface temperature of an object. Therefore, it is possible to use the intensity and wavelength of radiation emitted by an object to measure its surface temperature, without any physical contact. Objects at a particular temperature emit radiation over a range of wavelengths. The intensity of radiation emitted by an object, as a function of wavelength and surface temperature [13], can be written as

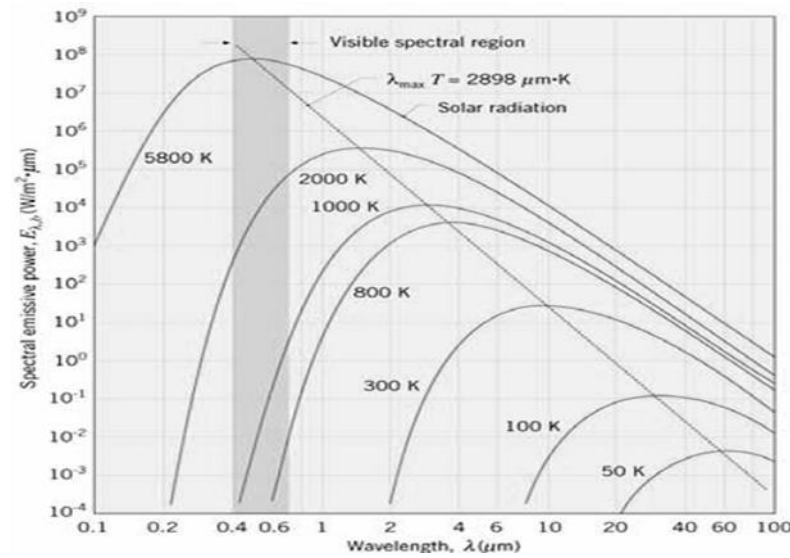
$$E_{\lambda} = \frac{A}{\lambda^5 (e^{(B/\lambda T)} - 1)} \quad (1)$$

where,

$E_{\lambda}$  = intensity of emitted radiation (W) at any particular wavelength (in m)

$T$  = surface temperature (K),  $A = 3.742 \times 10^8 \text{ W} \cdot \mu\text{m}^4 \cdot \text{m}^{-2}$  and  $B = 1.439 \times 10^4 \mu\text{m} \cdot \text{K}$  [12].

Based on (1), a series of curves are obtained which represents the amount of radiation emitted at each wavelength, for objects with different surface temperature as given in Fig. 2.7.



**Fig.2.7 Black body spectral radiant emittance.**

The wavelength, at which the intensity of emitted radiation is maximum, depends on the surface temperature. The higher the temperature, the shorter the wavelength at which most of the radiation is emitted and this relationship is described by Wien's displacement law as

$$\lambda_{\max} = 2.9/T$$

where,

$\lambda_{\max}$  = peak wavelength of emission in mm

T = surface temperature in K

The total radiation emitted by a body ( $E_{\text{Total}}$ ) can be obtained by integrating (1) across the whole wavelength range. According to Stefan-Boltzmann law, this total rate of emission per unit surface area is given by,

$$E_{\text{Total}} = \sigma T^4 \quad (3)$$

Where T is the absolute temperature in Kelvin and the Stefan-Boltzmann constant,  $\sigma = 5.67 \times 10^{-8} \text{ W/(m}^2\text{-K}^4\text{)}$ .

As the energy is released, the temperature decreases. This process continues until the target reaches thermal equilibrium with its environment. When radiation is incident on an object, portion of it is transmitted, some portion absorbed, and some reflected. For thermal equilibrium the total flux (measured in watts) must be constant and is defined as,

$$\Phi_{\text{Transmitted}} + \Phi_{\text{Absorbed}} + \Phi_{\text{Reflected}} = \Phi_{\text{Incident}} \quad (4)$$

The emissivity of a black body is equal to 1.0 and constant at all wavelengths and a body for which emissivity is less than 1.0, but still constant at all wavelengths, is known as a gray body. However, in real life most bodies are neither grey nor black and the emissivity is dependent on the wavelength of radiation being considered. The surface characteristics which govern emissivity also control the ability of a material to absorb radiation. For any body in thermal equilibrium, the emitted energy must be balanced by the energy absorbed from the surroundings. According to Kirchhoff's law, the absorptivity of radiation at any wavelength is equal to the emissivity of the material at the same wavelength and can be defined by the ratio as

$$\alpha = \frac{E_{\lambda}}{E_{b\lambda}} = \varepsilon \quad (5)$$

where  $\alpha$  and  $\varepsilon$  are the absorptivity and emissivity respectively,  $E_{\lambda}$  is the surface emissive power of a body and  $E_{b\lambda}$  is the surface emissive power of a black body. For real surfaces, during thermal equilibrium the transmissivity of solid surfaces is equal to zero, therefore (4) can be rewritten as

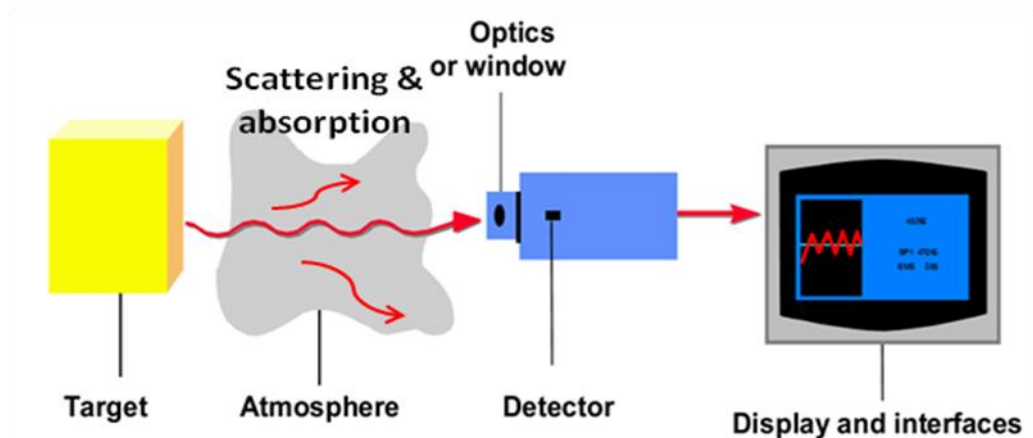
$$\Phi_{Absorbed} + \Phi_{Reflected} = \Phi_{Incident} \quad (6)$$

In case of ores and minerals in unpolished condition, the amount of heat reflected is very low; hence most of the heat incident on the ores is absorbed by them. The heat radiated by the uniformly heated ores is captured and displayed as thermal image through IR Thermography. The ores having low thermal absorptivity are heated up less and can be identified from the thermal images of the ore specimens [14].

#### 2.4.2 IR Measuring System

Every object above absolute zero temperature emits electromagnetic radiations of varying wavelength which are related to surface temperature. All the objects are composed of continually vibrating atoms, with higher energy atoms vibrating more frequently. The vibration of all charged particles, including these atoms, generates electromagnetic waves. Higher the temperature of an object, faster the vibration, and thus higher is the spectral radiant emittance from the object.

IR applications require radiation transmission through air, but the process of scattering and absorption attenuates the radiation as shown in Fig. 2.8.



**Fig. 2.8 IR measuring system.**

IR Thermographic measurement are typically done in one of the two spectral bands which exhibit relatively low absorption of light by water vapor and carbon dioxide; these are 3-5  $\mu\text{m}$  or 8-12  $\mu\text{m}$  band depending on the spectral sensitivity range of the detector used and are sometimes called “Atmospheric Windows”. The IR radiations are perceived by array of detectors which convert them into electric signals to be visualized on a display for analysis.

### 2.4.3 Modes of Excitation

IR Thermography is deployed by two approaches; passive and active.

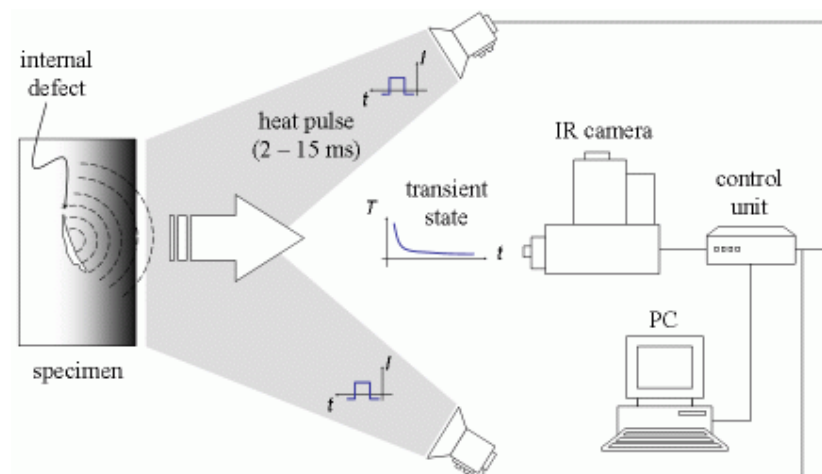
#### I. Passive Thermography

This approach tests the materials and structures which are naturally at different (often higher) temperature than the ambient. In most situations, passive thermography is used for qualitative assessment, given that the objective is to detect irregularities of the kind function or malfunction. Important applications of passive thermography are in predictive maintenance, military reconnaissance, medical imaging, forest fire detection, building thermal efficiency survey, road traffic monitoring, gas detection, agriculture and biology, etc.

#### II. Active Thermography

In this approach an external stimulus is necessary to induce relevant thermal contrast which is obtained by following methods:-

➤ **Pulse Thermography:** This is one of the well-known thermal stimulation methods in IR Thermography. This method is extensively used for thermographic testing due to the quickness of the inspection relying on a thermal stimulation pulse, with duration going from a few ms for high thermal conductivity material inspection to a few seconds for low thermal conductivity specimens. Such quick thermal stimulation allows direct deployment on the plant floor with convenient heating sources. Moreover, the brief heating prevents damage to the component. This method consists of briefly heating the specimen and then recording the temperature decay curve as shown in Fig. 2.9.



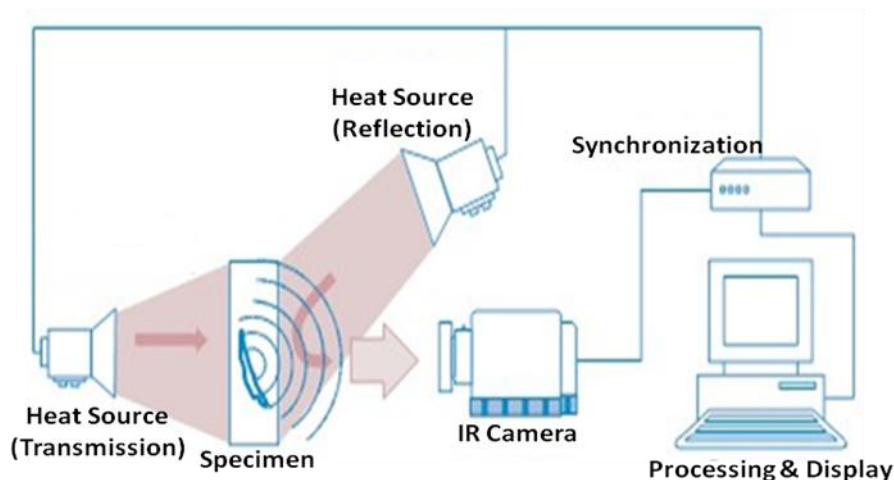
**Fig. 2.9 Basic Principle of Pulse Thermography.**

Various modes of excitation used in pulse thermography are:

1. Point Inspection: It involves heating with a laser or a focused light beam.
2. Line Inspection: It involves excitation using line lamps, heated wire, scanning laser, line of air jets (cool or hot), etc.
3. Surface Inspection: It involves heating using lamps, flash lamps, scanning laser, microwaves, etc. In case of microwave heating, direct internal heating of the part is achieved and since travel time is reduced from subsurface defect to surface with respect to surface to subsurface defect to surface heating, defects are delineated with better thermal contrast.

Possible observation methods are as follows:

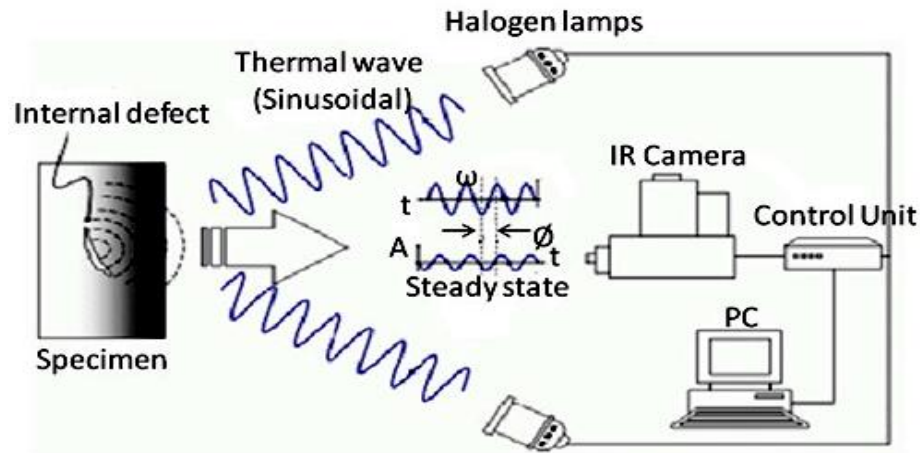
- a) Reflection- The thermal source and detector are located on the same side of the inspected component as shown in Fig. 2.10. This approach is used for detection of defects located close to the heated surface.
- b) Transmission- The heating source and detector are located on each side of the component. This approach allows detecting defect close to the rear surface because of the spreading effect of the thermal front.



**Fig. 2.10 Experimental set-up for Active Thermography inspection by Transmission and Reflection.**

- **Lock-in Thermography (LT):-** In lock-in Thermography, also known as Modulated Thermography, the specimen is stimulated with a periodic energy source. Typically, sinusoidal waves are used, although it is possible to use other periodic waveforms. When the input energy wave penetrates the object's surface, it is absorbed and phase shifted. When the input wave reaches areas within the object where the thermo-physical properties are not homogeneous in relation to the

surrounding material, (i.e. at delaminations or inclusions), the input wave is partially reflected as shown in Fig. 2.11.



**Fig. 2.11 Experimental set-up example for Lock-in Thermography.**

The reflected portion of the wave interferes with the incoming input wave at the surface of the object, causing an interference pattern in the local surface temperature, which oscillates at the same frequency as the thermal wave.

The internal structure of the object being examined can then be derived by evaluating the phase shift of the local surface temperatures in relation to the input energy wave. **Vibrothermography (VT):** - This technique makes use of mechanical oscillations, for example, a sonic or ultrasonic transducer that is in contact with the specimen as shown in Fig. 2.12. The defects are stimulated internally and the mechanical oscillations transmitted into the specimen which spread in all directions inside it. The mechanical energy is dissipated at defect locations such as cracks and delaminations in the form of heat waves that travel to the surface by conduction.

## CHAPTER 1

### INTRODUCTION

#### 1.1 BACKGROUND

The increasing demand and use of energy internationally, especially in India is heavily dependent on coal. India has some of the largest coal reserves in the world (approx. 267 billion tones). The energy derived from coal in India is about twice that of energy derived from oil, whereas worldwide, energy derived from coal is about 30% less than

energy derived from oil. Indian coal is of mostly sub-bituminous rank, followed by bituminous and lignite (brown coal). The mineral deposits of India are shown in the Fig. 1.1.



**Fig. 1.1 Mineral deposits of India**

Coal is the world's most abundant and widely distributed fossil fuel. It is a global industry that makes a significant economic contribution to the global economy. Coal is mined commercially in more than 50 countries and used in more than 70. Annual world coal consumption is about 5,800 million tons, of which about 75% is used for electricity production. This consumption is projected to nearly double by the year 2030 to meet the challenge of sustainable development and a growing demand for energy. Coal produces 40% of the world's electricity, which is double the share of its



nearest competitors (gas and hydro) and coal is an essential element in over 65% of the world's steel production [1].

The International Energy Agency (IEA) predicts that world energy demand will grow around 60% over the next 30 years, most of it in developing countries. China and India are very large countries in terms of both population and land mass, and both have substantial quantities of coal reserves[2]. Together, they account for 70% of the projected increase in world coal consumption. Strong economic growth is projected for both countries (averaging 6% per year in China and 5.4% per year in India from 2003 to 2030), and much of the increase in their demand for energy, particularly in the industrial and electricity sectors, is expected to be met by coal.

Coal is a complex combination of organic matter and inorganic mineral matter formed over year from successive layers of fallen vegetation [3]. Coals are classified by rank according to their progressive alteration in the natural metamorphosis from lignite to anthracite. Coal rank depends on the volatile matter, fixed carbon, inherent moisture, and oxygen; although no single parameter defines a rank. Typically, coal rank increases as the amount of fixed carbon increases and the amount of ash and moisture decreases.

Chemical composition of the coal is defined in terms of its proximate and ultimate (elemental) analysis. The parameters of proximate analysis are moisture, volatile matter, ash, and fixed carbon. Elemental or Ultimate analysis encompasses the quantitative determination of carbon, hydrogen, nitrogen, sulfur, and oxygen. The calorific value  $Q$ , of coal is the heat liberated by its complete combustion with oxygen.  $Q$  is a complex function of the elemental composition of the coal. Gross Calorific value  $Q$  is mostly determined by experimental measurements. A close estimate can be made with the Dulong formula:

$$Q = (144.4 \%[C]) + (610.2 \%[H]) - (65.9 \%[O]) + (0.39 \%[O]^2)$$

$Q$  is given in Kcal/kg or Btu/lb.

Indian coal is classified by grades defined on the basis of Useful Heat Value (UHV). UHV is an expression derived from ash and moisture contents for coals as per the Government of India notification. The ultimate analysis of coal used in power plants in India is readily not available. Ultimate analysis of D, E, and F grade coal is

provided by Central Fuel Research Laboratory (CFRI), Jharkhand, India. Ultimate analysis of coal used at Dadri, Rihand, Singrauli, Chandrapur, and Dahanu power plants is obtained from National Energy Technology Laboratory, Pittsburgh, USA. The Neyveli Lignite Corporation provided by personal communication, the ultimate analysis for lignite is used at the Neyveli and Kutch power plants. These are presented in Tables 1.1 and 1.2.

**Table 1.1 Elemental analysis, moisture content, and grades of typical Indian coals**

<b>Coal Grade</b>	<b>C(%)</b>	<b>H(%)</b>	<b>N<sub>2</sub>(%)</b>	<b>O<sub>2</sub>(%)</b>	<b>A(%)</b>	<b>M(%)</b>	<b>NCV (Kcal/Kg)</b>	<b>UHV (cal/gm)</b>
D	33.1	2.46	0.83	NA	25.9	7.2	4999.0	4332.0
D	30	2.48	0.69	NA	27.1	2.9	5555.0	4760.0
D	32.31	2.12	0.78	NA	25	7.3	5068.0	4442.0
E	37.9	2.4	0.8	6%	30.4	7.5	4529.0	3670.0
F1	41.87	3.33	0.94	6%	34.07	7.8	4137.0	3122.0
F2	44.47	3.37	0.99	6%	36.3	8.4	3833.0	2731.0

**Table 1.2 Elemental analysis and moisture content of the coal used at the seven power plants in India**

<b>Coal</b>	<b>C(%)</b>	<b>H(%)</b>	<b>S(%)</b>	<b>N<sub>2</sub>(%)</b>	<b>O<sub>2</sub>(%)</b>	<b>A(%)</b>	<b>M(%)</b>	<b>NCV</b>
Dadri	40.3	4.16	0.5	0.9	15.92	38.22	NA	NA
Rihand	37.74	3.26	0.39	0.73	14.65	43.23	NA	NA
Singrauli	50.22	4.78	0.33	1.09	17.25	26.33	NA	NA
Chandrapur	37.69	2.66	0.8	1.07	5.78	47.0	5	3649.9
Dahanu	42.39.0	3.73	0.39	0.82	14.21	38.46	5.93	3986.37
Neyveli Lignite	26.09	2.33	1.5	0.24	16.33	7.0	47	2229.0
Kutch Lignite	28.33	3.03	2.25	0.88	13.94	15.0	36	2900.0

NA: Not Available

C: Carbon

H: Hydrogen

S: Sulfur

N: Nitrogen

CV: Calorific value

A: Ash

M: Moisture

UHV: Useful heat value =  $8900 - 138(A+M)$

GCV: Gross Calorific Value =  $(UHV + 3645 - 75.4 M)/1.466$

NCV: Net Calorific Value =  $GCV - 10.02M$

Relationship of GCV, UHV, and NCV is empirical.

Ultimate analysis of typical United States coals is given in Table 1.3 for the sake of comparison.

**Table 1.3 Ultimate analysis of typical US coals**

		<b>Chandrapur (India)</b>	<b>Ohio (USA)</b>
1	Fixed carbon	27.5%	44.0
2	Total carbon	37.69%	64.2%
3	Hydrogen	2.66%	5.0 %
4	Nitrogen	1.07%	1.3 %
5	Oxygen (difference)	5.78%	11.8 %
6	Sulfur	0.8%	1.8 %
7	Ash	47%	16%
8	Total moisture	5%	2.8
9	Gross calorific value Kcal/Kg	3400	6378
10	Coal per unit of electricity (Kg/KWH)	0.77	0.36

The coal properties including calorific values differ depending upon the colliery. The calorific value of the Indian coal (~15 MJ/kg) is less than the normal range of 21 to 33 MJ/Kg (gross).

Coal as a fossil fuel is used to supply heat energy in many power plants, boilers and furnaces in various industries all over the world. Coal ash is the oxidized residue left after burning coal and is an important factor in determining the calorific value of coal. Coal ash consists mainly of alumina, silica and iron oxide with small quantities of many other metal oxides.

Even as demand grows, society expects cleaner energy with less pollution and an increasing emphasis on environmental sustainability. The coal industry recognizes it must meet the challenge of environmental sustainability; in particular it must reduce its greenhouse gas emissions if it is to remain a part of a sustainable energy future.

The ash content in Indian coal ranges from 25% to 75%. In power plants, when the ash content increases from 6% to 75%, the adverse effects on the system are as follows[5]:

- Total boiler area requirement increases by 69%.
- Efficiency of boiler fans drop by 22%–27%.
- Induced draft (ID), forced draft (FD), and primary air (PA) fans show an increase in specific energy consumption (SEC) of around 30%, 6%–14%, and 2%–7% respectively.
- Drum mills show an increase in SEC of 115% while ball-race mills and bowl mills show an increase in SEC of 30%.
- The gross and net overall efficiencies are reduced to 77% and 66% of their original values.
- The ratio of the specific fuel consumption at a given ash content to that at standard ash of 6% increases from 1 to 10.
- Specific fuel consumption (SFC) gross increases from 0.35 to 3.0.

Determination of ash and moisture present in coals, by chemical analysis method is time consuming and one cannot have an on-line analysis. This absence of real-time online analysis data creates problems for plant operators and quality assurance managers. Hence an alternate technique is needed to study the quality of coals in terms of ash and moisture present in them and henceforth find the suitability of specific coals for individual combustion systems.

## **1.2 OBJECTIVE**

The major objectives of this project are:-

- To provide a faster way to determine the ash and moisture content in coal and thereby to find the suitability of specific coals for individual combustion systems.
- To develop a software for obtaining the calibration curve and henceforth predict ash and moisture in the coal samples.

### **1.3 PLAN OF WORK**

- Collection of coal samples from different mines.
- Optimization of heating source and heating time for IR thermography of coal samples.
- IR image capturing and analysis.
- Chemical analysis of coal.
- Development of Calibration Curve for determination of ash and moisture content in coal.
- Software development for prediction of ash and moisture content in coal.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 LIMITATIONS OF PRESENT METHOD**

Standard test methods for routine coal analysis include those mentioned in American Society for Testing and Materials (ASTM) and, with limited application, those of the International Organization of Standardization (ISO)[6]. In analyzing coal research samples for their chemical composition, it is apparent that certain current standard test methods require modification or they are not applicable. Coal research, and possibly most new uses of coal, requires higher standards of quality control and accuracy than that are currently quoted in many of the existing standard test methods. These factors become increasingly more important when economic decisions have to be made based on the validity, i.e., accuracy, of coal analysis data. The problems related with ash and moisture analysis are given below.

##### **2.1.3 Limitations of Moisture Analysis**

Determining the moisture in the analysis by weight loss at 104-110°C presents several problems, especially if the current ASTM method D3173 (1) is used. The ASTM method is unsuitable for low-rank coals because it does not use an inert gas as the purge gas and recommends too short a drying period. Due to oxidation gain in weight can take place as the sample reacts with oxygen in the air and not all of the moisture is removed during the recommended one-hour drying time at 104-110°C heating. In addition, with certain low-rank coals, decarboxylation during the drying period can result in weight loss.

##### **2.1.4 Limitations of Ash Analysis**

The ash in coal is the non-combustible residue that remains when coal is burned. In the ASTM method D3174 (3), the coal sample is placed in a cold furnace and heated gradually so the temperature reaches 450 to 500°C in one hour and 700 to 750°C at the end of the second hour. The ISO method 1171 (4) recommends 815°C final temperature. In both methods the sample is ignited at the appropriate final temperature to constant weight. Furthermore, there is difference of opinion as to the proper rate of

heating required for the determination of ash in coals high in calcite and pyrite[7]. In the determination of ash, particularly in coals containing appreciable amounts of pyrite and calcite, there is danger that the ash will retain indefinite amounts of sulfur which causes erratic results. Although it is widely recognized that the rate of heating influences the sulfur retention, there is disagreement as to how fast the heating rate may be and still give reliable test results.

Therefore, a relatively capital inexpensive, reliable online sensing system, which is also faster compared to the methods currently in use, is desirable for the compositional analysis of coals.

### 2.3 COAL CLASSIFICATION AND ANALYSIS

Coal is classified into three major types namely anthracite, bituminous, and lignite. However there is no clear demarcation between them and coal is also further classified as semi- anthracite, semi-bituminous, and sub-bituminous[8]. Anthracite is the oldest coal from geological perspective. It is a hard coal composed mainly of carbon with little volatile content and practically no moisture. Lignite is the youngest coal from geological perspective. It is a soft coal composed mainly of volatile matter and moisture content with low fixed carbon. Fixed carbon refers to carbon in its free state, not combined with other elements. Volatile matter refers to those combustible constituents of coal that vaporize when coal is heated. The common coals used in Indian industry are bituminous and sub-bituminous coal. The gradation of Indian coal based on its calorific value is given in the table 2.1

**Table 2.1 Gradation of Indian coal**

Grade	Calorific Value Range ( in kCal/kg)
A	Exceeding 6200
B	5600 – 6200
C	4940 – 5600
D	4200 – 4940
E	3360 – 4200
F	2400 – 3360
G	1300 – 2400

Normally D, E and F coal grades are available to Indian Industry.

### **2.3.1 Analysis of Coal**

Analysis of coal is mostly done by two methods: ultimate analysis and proximate analysis[9]. The ultimate analysis is used to determine all coal constituent elements, solid or gaseous while the proximate analysis technique determines the fixed carbon, volatile matter, moisture and ash percentages. The ultimate analysis is carried out in a properly equipped laboratory by a skilled chemist, while proximate analysis can be done with a simple apparatus. It may be noted that proximate has no connection with the word “approximate”.

### **2.3.2 Proximate Analysis**

Proximate analysis indicates the percentage by weight of the Fixed Carbon, Volatiles, Ash, and Moisture Content in coal[10]. The amounts of fixed carbon and volatile combustible matter directly contribute to the heating value of coal. Fixed carbon acts as a main heat generator during burning. High volatile matter content indicates easy ignition of fuel. The ash content is important in the design of the furnace grate, combustion volume, pollution control equipment and ash handling systems of a furnace.

#### **(iv) Measurement of Moisture**

Determination of moisture is carried out by placing a sample of powdered raw coal of size 200-micron size in an uncovered crucible and it is placed in the oven kept at  $108 \pm 2$  °C along with the lid. Then the sample is cooled to room temperature and weighed again. The loss in weight represents moisture.

#### **(v) Measurement of Volatile Matter**

Fresh sample of crushed coal is weighed, placed in a covered crucible, and heated in a furnace at  $900 \pm 15$  °C. The sample is cooled and weighed. Loss of weight represents moisture and volatile matter. The remainder is coke (fixed carbon and ash).

#### **(vi) Measurement of Carbon and Ash**

The cover from the crucible used in the last test is removed and the crucible is heated over the Bunsen burner until all the carbon is burned. The residue is weighed, which is the incombustible ash. The difference in weight from the previous weighing is the fixed carbon. In actual practice Fixed Carbon or FC is derived by subtracting from 100 the value of moisture, volatile matter and ash.

A typical proximate analysis of various grades of coal is given in the Table 2.2.



<b>TABLE 2.2 TYPICAL PROXIMATE ANALYSIS OF VARIOUS COALS (IN PERCENTAGE)</b>			
<b>Parameter</b>	<b>Indian Coal</b>	<b>Indonesian Coal</b>	<b>South African Coal</b>
Moisture	5.98	9.43	8.5
Ash	38.63	13.99	17
Volatile matter	20.70	29.79	23.28
Fixed Carbon	34.69	46.79	51.23

### **2.2.3 Significance of Various Parameters in Proximate Analysis**

#### **(a) Fixed carbon:**

Fixed carbon is the solid fuel left in the furnace after volatile matter is distilled off. It consists mostly of carbon but also contains some hydrogen, oxygen, sulphur and nitrogen not driven off with the gases. Fixed carbon gives a rough estimate of heating value of coal.

#### **(b) Volatile Matter:**

Volatile matters include methane, hydrocarbons, hydrogen and carbon monoxide, and incombustible gases like carbon dioxide and nitrogen found in coal. Thus the volatile matter is an index of the gaseous fuels present. Typical range of volatile matter is 20 to 35%.

Volatile Matter

- Proportionately increases flame length, and helps in easier ignition of coal.
- Sets minimum limit on the furnace height and volume.
- Influences secondary air requirement and distribution aspects.
- Influences secondary oil support

#### **(c) Ash Content:**

Ash is an impurity that will not burn. Typical range is 5 to 40%.

Ash

- Reduces handling and burning capacity of coal
- Increases handling costs.
- Affects combustion efficiency and boiler efficiency
- Causes clinkering and slagging.

#### **(d) Moisture Content:**

Moisture in coal must be transported, handled and stored. Since it replaces combustible matter, it decreases the heat content per kg of coal. Typical range is 0.5 to 10%

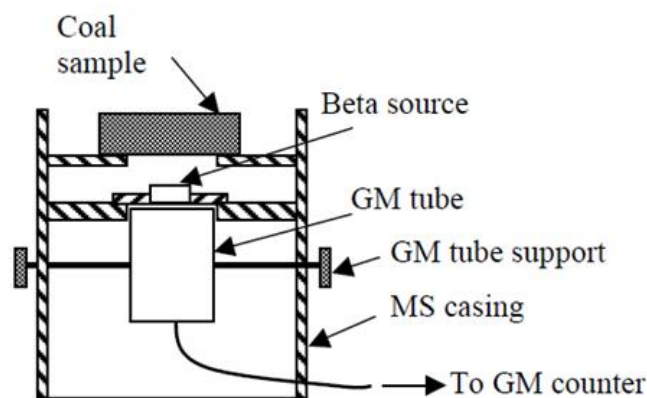
Moisture

- Increases heat loss, due to evaporation and superheating of vapour
- Helps, to a limit, in binding fines.
- Aids radiation heat transfer.

### **2.3 TECHNIQUES USED FOR DETERMINATION OF ASH IN COAL**

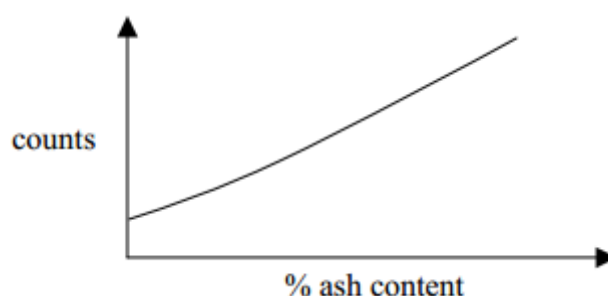
#### **2.3.1 Measurement of ash content in coal using beta ray back scattering technique**

Method of on-line measurement of ash content in coal by back scattering is based on the difference in atomic number ( $Z$ ) of coal and ash constituents. The atomic number of elements O, Al, Si, Fe of which ash is essentially composed, differ quite extensively from the atomic number of carbon and therefore those interactions of radiation with matter, which depend upon the atomic number can be utilized to determine the ash percentage in coal. The components of the coal (H, C, N, O) have lower atomic number than those of ash (Mg, Al, Si, S, Ca, Fe). This makes it a two component system. The intensity of the reflected beam with the same ash composition is a function of the ash content. The set up for beta backscattering for ash measurement in coal is shown in the Fig. 2.1.



**Fig. 2.1 Beta backscattering set up for ash measurement in coal**

For the ash analysis one would choose a high energy  $\beta$  source with a fairly large half life to minimize the error due to decay. Activity should be high enough to give required count in reasonable time limit. The ideal selection is Sr90-Y90. (half life = 28 years,  $E_{\max} = 2.2$  Mev and of activity 4  $\mu$  curie.) GM tube is used as the detector with the necessary counting system. The graph between ash content and count rate is given in the Fig. 2.2. The operating voltage of the detector has to be within the plateau region. Various coal samples of known ash content are prepared. The thickness of the sample should be more than the saturation thickness to ensure that the back scattered intensity is independent of the thickness of the sample.



**Fig. 2.2 Plot of ash content v/s count rate**

### **2.3.2 Proximate Analysis of Coal and Coke using the STA 8000 Simultaneous Thermal Analyzer**

Proximate analysis has long been used to determine the rank of coals by separating volatile components, fixed carbon and inert components. Because of the wide ranging quality of coal products and the commercial value of ranking these products the need for good methods is obvious. To meet these needs there are ASTM tests to perform these separations separately using specialized industrial equipment. The STA8000 Simultaneous Thermal Analyzer (STA) is able to analyze coal and coke to obtain Proximate Analysis data of volatiles, fixed carbon and ash using 10 to 100 milligram samples[12].

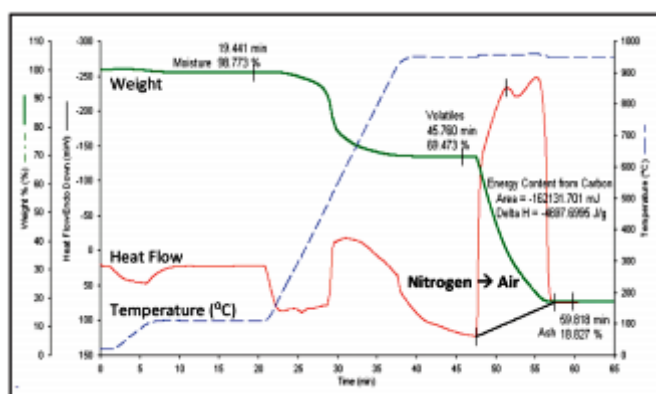
The instrument requirements needed are the ability to accurately record the weight of a sample as it is heated over a temperature range and held isothermally at designated temperatures, then change the sample's environmental atmosphere from inert to oxidizing. The PerkinElmer STA 8000 which is shown in the Fig. 2.3 easily meets these requirements, including accurate temperature and gas control, a balance with microgram sensitivity, and software to facilitate and automate the analysis.



**Fig. 2.3 The STA 8000 Simultaneous Thermal Analyzer.**

The weighing mechanism is ideally suited for routine quality testing use. The STA 8000 uses a top loading balance. This makes it easier to load, and when loading there is no force applied to a long balance arm or hang-down wire that could result in damage to the balance system. The sample is weighed and loaded at 25 °C, heating to 110 °C, holding for 10 minutes then heating to 950 °C where the temperature was held until constant weight was achieved in nitrogen. Then the sample atmosphere was switched to air (somewhat diluted by the balance purge). When the weight was again constant after the combustion of the fixed carbon component the analysis was ended and the apparatus cooled for the next analysis.

For determination of total volatiles the sample is heated from 107 °C to 950 °C and held isothermally to drive off all volatile components. After the switch to an oxidizing atmosphere combustion at 950 °C is rapid and complete and the weight loss gives the fixed carbon. After combustion, the remaining residue is the ash content of the coal, which can be read directly from the weight. The proximate analysis using STA 8000 is shown in the Fig. 2.4.

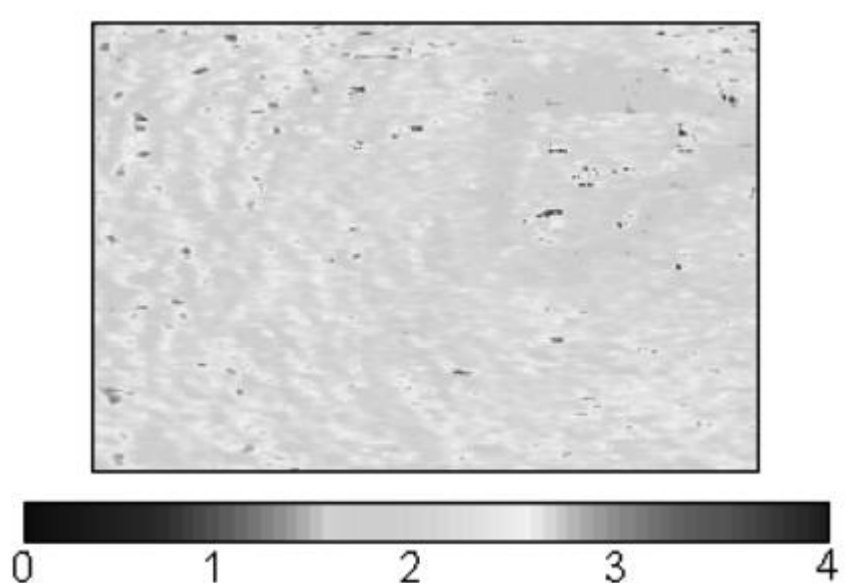


**Fig. 2.4 Proximate analysis of coal using the STA 8000.**

### 2.3.3 Real-time coal or ore grade sensor

A real-time coal content or ore grade sensor can be used during exploration, mining, and processing operations. The sensor is developed based on advanced multi-spectral and hyper-spectral imaging technology that is currently used in airborne and satellite applications.

This project utilizes the unique spectral characteristics of coal and platinum or palladium to quantify coal content and ore grade. Because the instrument will utilize imaging technology that requires only a few hundredths of a second per measurement, the mineral content across a working surface or in particulate matter will be rapidly measured. Using modern reflectance spectroscopy and digital image processing techniques, the spectral signatures of target minerals will be used to generate and easily interpret a false color map indicating mineral content in coal or ore. This will allow for greater selectivity as well as decrease environmental impacts and energy requirements in exploration, mining, and processing activities. The processed microspectral image of ore sample is shown in the Fig.2.5.



**Fig. 2.5 Processed microspectral image of ore sample**

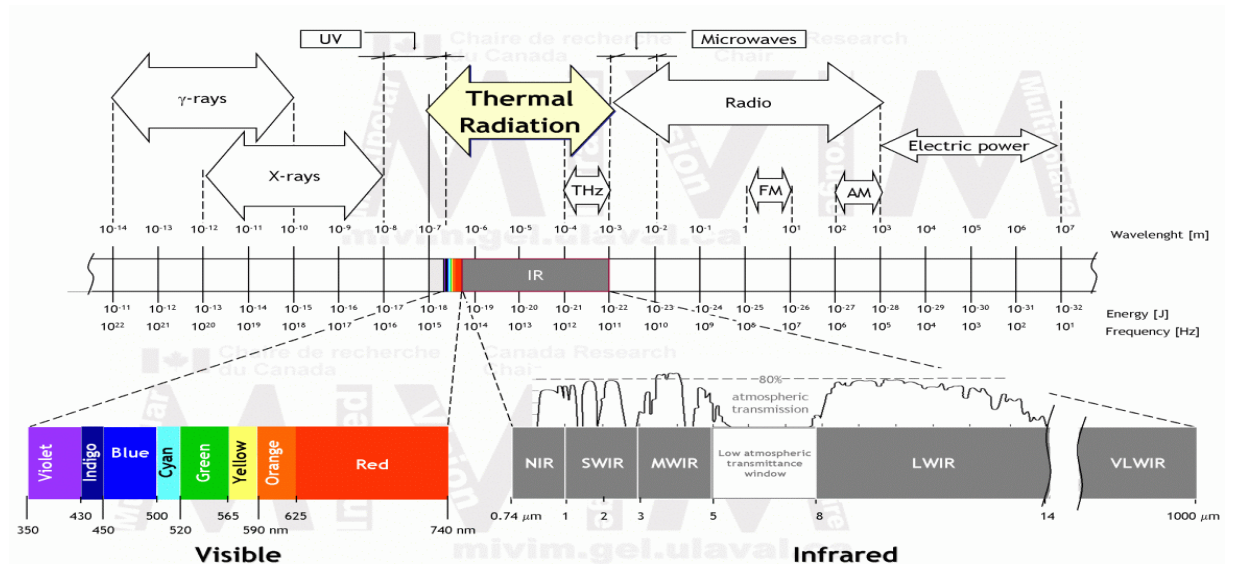
The dark areas above show false colouring used to highlight the presence of sulfides within the ore.

It is evident from the literature study that IR Thermography based techniques have not yet been explored for compositional analysis of coal. IR Thermography has been found to be a useful technique for identification and gradation of alumina-rich iron ores [15] and this technique may be further explored for compositional analysis of

coal. As this technique is based on the relative difference in thermal absorptivity of ore constituents for studying their quality, this can also be used to determine the ash and moisture content in coal. Therefore, the prime objective of this project is to develop an IR thermography based non-invasive technique for faster compositional analysis of coal.

## 2.5 INFRARED THERMOGRAPHY

All objects with surface temperature above absolute zero emit electromagnetic radiation that is uniquely related to object temperature. There is no fundamental difference between radiations in the different bands of the electromagnetic spectrum. They are all governed by same laws and the only difference is due to the variation in wavelength as shown in Fig. 2.6 of the electromagnetic spectrum.



**Fig. 2.6 Electromagnetic Spectrum.**

Infrared Thermography is the science of acquisition and analysis of thermal information by using non-contact thermal imaging devices. Infrared (IR) emission is electromagnetic radiation with longer wavelengths than those of visible light but shorter than microwaves. The infrared band spans from 0.76  $\mu\text{m}$  to 1000  $\mu\text{m}$ , also called “thermal band”. The infrared spectrum is further divided into 3 regions:-

- [4] Short wave/Near Infrared (0.76 - 2  $\mu\text{m}$ )
- [5] Medium wave/Middle Infrared (2 - 4  $\mu\text{m}$ )
- [6] Long wave/Far Infrared (4 - 1000  $\mu\text{m}$ )

Thermography is one of the widely used non-destructive test (NDT) techniques. Some of the advantages of IR Thermography are:

- It is a non-contact method of temperature measurement which allows collecting data from hazardous or physically inaccessible objects and does not intrude upon or affect the target at all.
- It has fast inspection rate (up to few m<sup>2</sup> at a time).
- It keeps the user safe since measurement is non-contact and no harmful radiations are involved.
- It also facilitates measurement of moving targets and enables capturing of fast changing thermal patterns in real time.
- There is no risk of contamination and no mechanical effect on the surface of the object; thus wear free. Lacquered surfaces are not scratched and soft surfaces can also be measured.
- The thermal imaging technique is being widely used in various fields which include: inspections for predictive maintenance, non-destructive evaluation of thermal and mechanical properties, building science, agriculture, military reconnaissance, weapons guidance and medical imaging.

As with all NDT methods, thermo graphic inspection also has its limitations, which include:

- Difficulty in obtaining a quick, uniform and highly energetic thermal stimulation over a large surface.
- Effects of thermal losses which induce spurious contrasts affecting the reliability of interpretation.
- It is a boundary technique which allows inspecting a limited thickness of material under the surface and detects defects which results in measurable change in thermal properties.
- The equipment cost is usually higher.

### **2.5.1 Governing Equations**

The electromagnetic radiation depends upon two parameters; its wavelength  $\lambda$  and intensity  $E$  and these two parameters affect the surface temperature of an object. Therefore, it is possible to use the intensity and wavelength of radiation emitted by an object to measure its surface temperature, without any physical contact. Objects at a particular temperature emit radiation over a range of wavelengths. The intensity of radiation emitted by an object, as a function of wavelength and surface temperature [13], can be written as

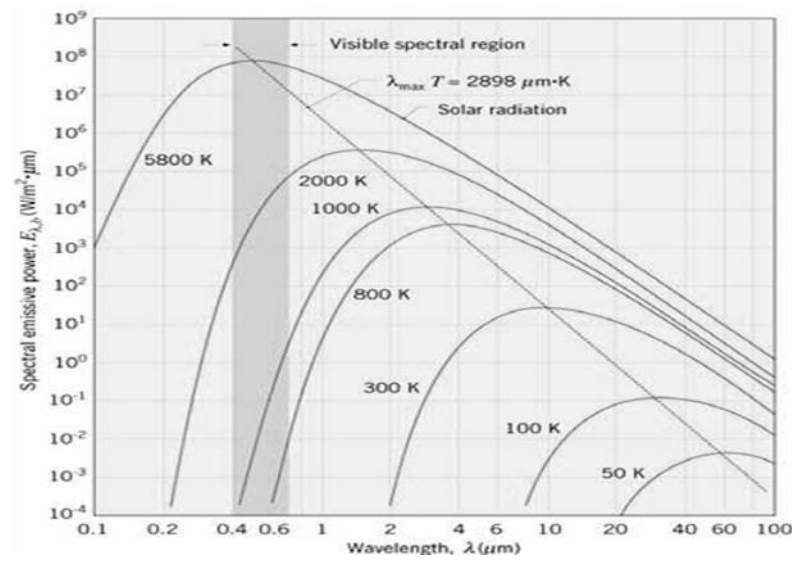
$$E_{\lambda} = \frac{A}{\lambda^5 (e^{(B/\lambda T)} - 1)} \quad (1)$$

where,

$E_{\lambda}$  = intensity of emitted radiation (W) at any particular wavelength (in m)

T = surface temperature (K),  $A = 3.742 \times 10^8 \text{ W} \cdot \mu\text{m}^4 \cdot \text{m}^{-2}$  and  $B = 1.439 \times 10^4 \mu\text{m} \cdot \text{K}$  [12].

Based on (1), a series of curves are obtained which represents the amount of radiation emitted at each wavelength, for objects with different surface temperature as given in Fig. 2.7.



**Fig.2.7 Black body spectral radiant emittance.**

The wavelength, at which the intensity of emitted radiation is maximum, depends on the surface temperature. The higher the temperature, the shorter the wavelength at which most of the radiation is emitted and this relationship is described by Wien's displacement law as

$$\lambda_{\max} = 2.9 / T$$

where,

$\lambda_{\max}$  = peak wavelength of emission in mm

T = surface temperature in K

The total radiation emitted by a body ( $E_{\text{Total}}$ ) can be obtained by integrating (1) across the whole wavelength range. According to Stefan-Boltzmann law, this total rate of emission per unit surface area is given by,

$$E_{\text{Total}} = \sigma T^4 \quad (3)$$



Where T is the absolute temperature in Kelvin and the Stefan-Boltzmann constant,  $\sigma = 5.67 \times 10^{-8} \text{ W/(m}^2\text{-K}^4\text{)}$ .

As the energy is released, the temperature decreases. This process continues until the target reaches thermal equilibrium with its environment. When radiation is incident on an object, portion of it is transmitted, some portion absorbed, and some reflected. For thermal equilibrium the total flux (measured in watts) must be constant and is defined as,

$$\Phi_{Transmitted} + \Phi_{Absorbed} + \Phi_{Reflected} = \Phi_{Incident} \quad (4)$$

The emissivity of a black body is equal to 1.0 and constant at all wavelengths and a body for which emissivity is less than 1.0, but still constant at all wavelengths, is known as a gray body. However, in real life most bodies are neither grey nor black and the emissivity is dependent on the wavelength of radiation being considered. The surface characteristics which govern emissivity also control the ability of a material to absorb radiation. For any body in thermal equilibrium, the emitted energy must be balanced by the energy absorbed from the surroundings. According to Kirchhoff's law, the absorptivity of radiation at any wavelength is equal to the emissivity of the material at the same wavelength and can be defined by the ratio as

$$\alpha = \frac{E_{\lambda}}{E_{b\lambda}} = \varepsilon \quad (5)$$

where  $\alpha$  and  $\varepsilon$  are the absorptivity and emissivity respectively,  $E_{\lambda}$  is the surface emissive power of a body and  $E_{b\lambda}$  is the surface emissive power of a black body. For real surfaces, during thermal equilibrium the transmissivity of solid surfaces is equal to zero, therefore (4) can be rewritten as

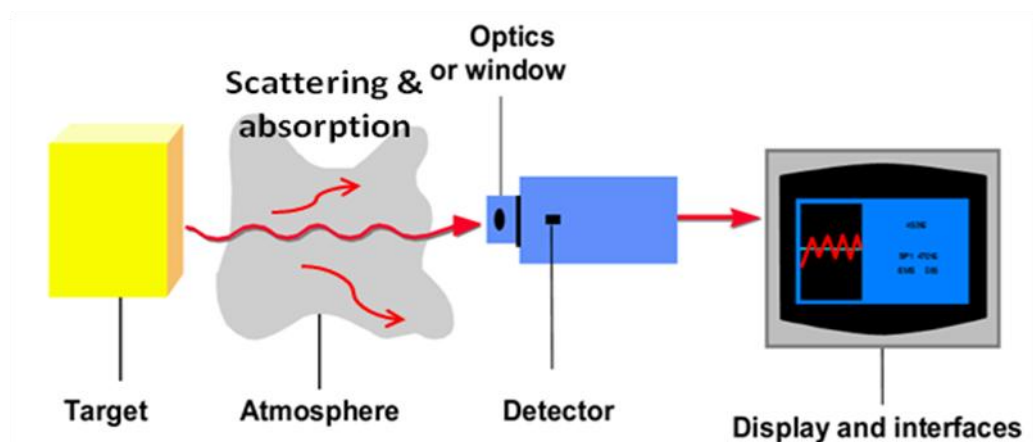
$$\Phi_{Absorbed} + \Phi_{Reflected} = \Phi_{Incident} \quad (6)$$

In case of ores and minerals in unpolished condition, the amount of heat reflected is very low; hence most of the heat incident on the ores is absorbed by them. The heat radiated by the uniformly heated ores is captured and displayed as thermal image through IR Thermography. The ores having low thermal absorptivity are heated up less and can be identified from the thermal images of the ore specimens [14].

### 2.5.2 IR Measuring System

Every object above absolute zero temperature emits electromagnetic radiations of varying wavelength which are related to surface temperature. All the objects are composed of continually vibrating atoms, with higher energy atoms vibrating more frequently. The vibration of all charged particles, including these atoms, generates electromagnetic waves. Higher the temperature of an object, faster the vibration, and thus higher is the spectral radiant emittance from the object.

IR applications require radiation transmission through air, but the process of scattering and absorption attenuates the radiation as shown in Fig. 2.8.



**Fig. 2.8 IR measuring system.**

IR Thermographic measurement are typically done in one of the two spectral bands which exhibit relatively low absorption of light by water vapor and carbon dioxide; these are 3-5  $\mu\text{m}$  or 8-12  $\mu\text{m}$  band depending on the spectral sensitivity range of the detector used and are sometimes called “Atmospheric Windows”. The IR radiations are perceived by array of detectors which convert them into electric signals to be visualized on a display for analysis.

### 2.5.3 Modes of Excitation

IR Thermography is deployed by two approaches; passive and active.

#### I. Passive Thermography

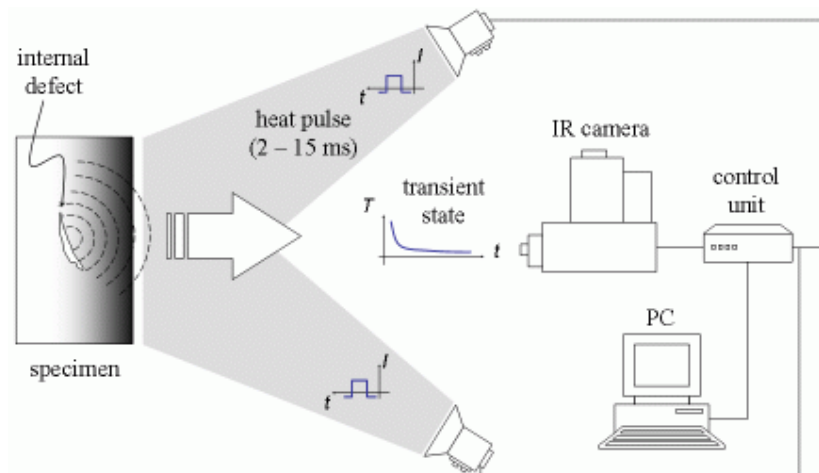
This approach tests the materials and structures which are naturally at different (often higher) temperature than the ambient. In most situations, passive thermography is used for qualitative assessment, given that the objective is to detect irregularities of

the kind function or malfunction. Important applications of passive thermography are in predictive maintenance, military reconnaissance, medical imaging, forest fire detection, building thermal efficiency survey, road traffic monitoring, gas detection, agriculture and biology, etc.

## II. Active Thermography

In this approach an external stimulus is necessary to induce relevant thermal contrast which is obtained by following methods:-

➤ **Pulse Thermography:** This is one of the well-known thermal stimulation methods in IR Thermography. This method is extensively used for thermographic testing due to the quickness of the inspection relying on a thermal stimulation pulse, with duration going from a few ms for high thermal conductivity material inspection to a few seconds for low thermal conductivity specimens. Such quick thermal stimulation allows direct deployment on the plant floor with convenient heating sources. Moreover, the brief heating prevents damage to the component. This method consists of briefly heating the specimen and then recording the temperature decay curve as shown in Fig. 2.9.



**Fig. 2.9 Basic Principle of Pulse Thermography.**

Various modes of excitation used in pulse thermography are:

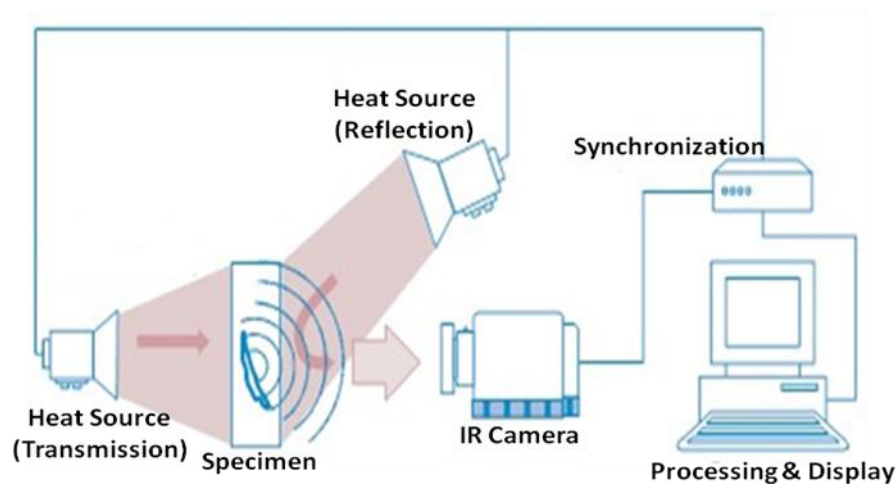
4. Point Inspection: It involves heating with a laser or a focused light beam.
5. Line Inspection: It involves excitation using line lamps, heated wire, scanning laser, line of air jets (cool or hot), etc.

[3] Surface Inspection: It involves heating using lamps, flash lamps, scanning laser, microwaves, etc. In case of microwave heating, direct internal heating of the part is achieved and since travel time is reduced from subsurface defect to surface with

respect to surface to subsurface defect to surface heating, defects are delineated with better thermal contrast.

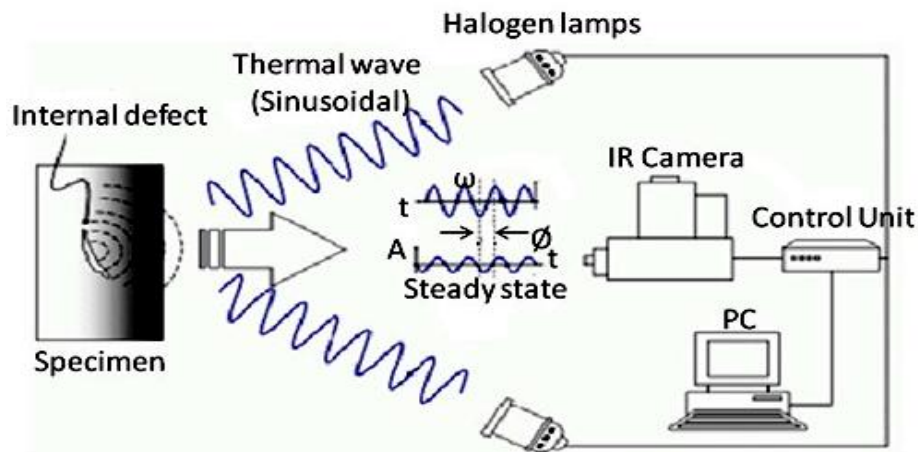
Possible observation methods are as follows:

- c) Reflection- The thermal source and detector are located on the same side of the inspected component as shown in Fig. 2.10. This approach is used for detection of defects located close to the heated surface.
- d) Transmission- The heating source and detector are located on each side of the component. This approach allows detecting defect close to the rear surface because of the spreading effect of the thermal front.



**Fig. 2.10 Experimental set-up for Active Thermography inspection by Transmission and Reflection.**

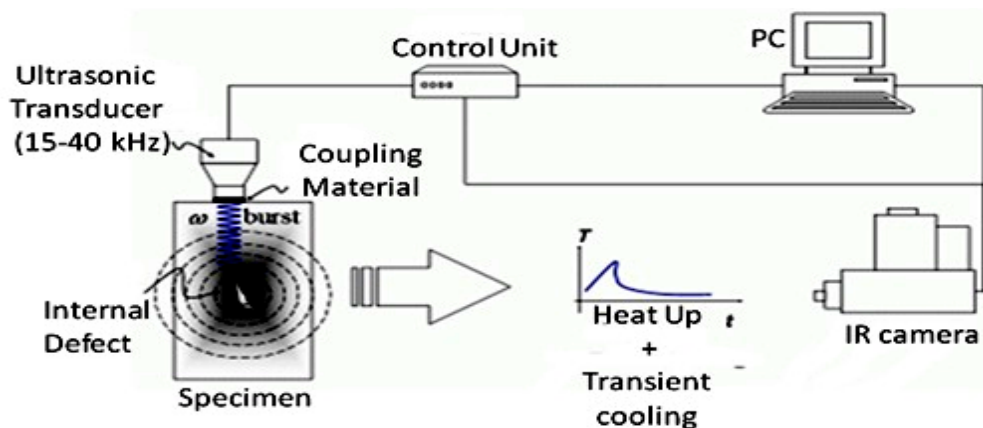
- **Lock-in Thermography (LT):-** In lock-in Thermography, also known as Modulated Thermography, the specimen is stimulated with a periodic energy source. Typically, sinusoidal waves are used, although it is possible to use other periodic waveforms. When the input energy wave penetrates the object's surface, it is absorbed and phase shifted. When the input wave reaches areas within the object where the thermo-physical properties are not homogeneous in relation to the surrounding material, (i.e. at delaminations or inclusions), the input wave is partially reflected as shown in Fig. 2.11.



**Fig. 2.11 Experimental set-up example for Lock-in Thermography.**

The reflected portion of the wave interferes with the incoming input wave at the surface of the object, causing an interference pattern in the local surface temperature, which oscillates at the same frequency as the thermal wave.

The internal structure of the object being examined can then be derived by evaluating the phase shift of the local surface temperatures in relation to the input energy wave. **Vibrothermography (VT):** - This technique makes use of mechanical oscillations, for example, a sonic or ultrasonic transducer that is in contact with the specimen as shown in Fig. 2.12. The defects are stimulated internally and the mechanical oscillations transmitted into the specimen which spread in all directions inside it. The mechanical energy is dissipated at defect locations such as cracks and delaminations in the form of heat waves that travel to the surface by conduction.



**Fig. 2.12 Experimental set-up for vibrothermography inspection by reflection mode.**

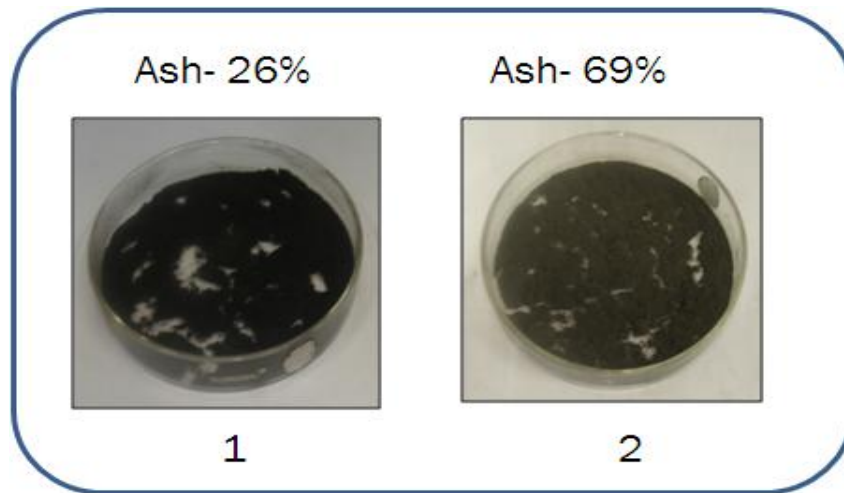
VT is more suitable for relatively small objects and detecting micro cracks. The most inconvenient aspect of VT is the need of a coupling media between the sample and the transducer, and the need of holding the specimen. On the other hand, there is only minimal heating of the inspected specimen since energy is usually dissipated mostly at the defective areas, although there is some localized heating at the coupling and clamping points.

## **CHAPTER 3**

### **METHODOLOGY**

#### **3.1 Coal Sample preparation for IR imaging**

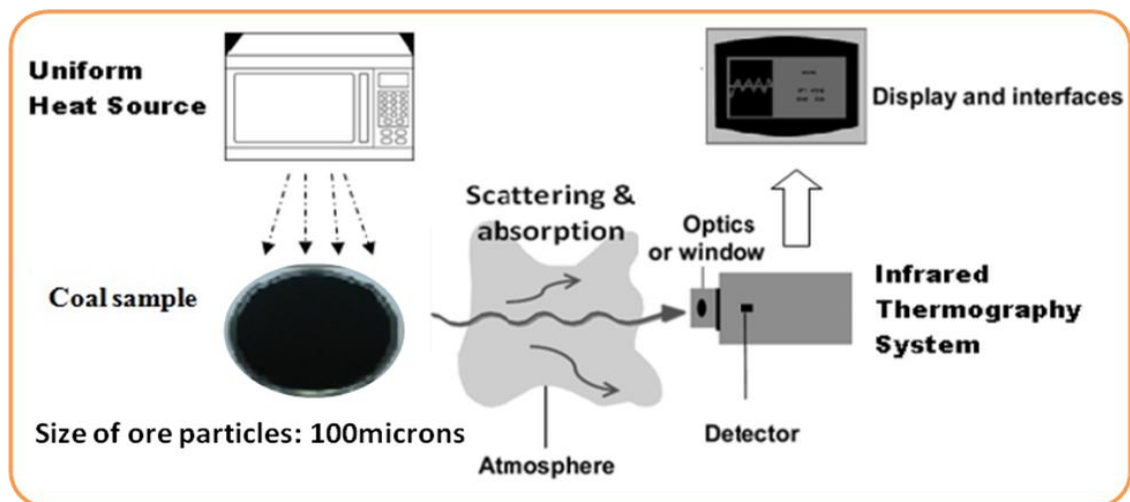
Different coal samples with ash content varying in the range of 25.0 to 85.0 wt.% and moisture content from 1.0 to 6.0 wt.% have been collected from Coal Characterization Centre in CSIR-NML. Four different sets of samples were collected in which the first two sets were from Sambaleswari block and last two sets from Talcher block. These samples were crushed to fine powder having particle size of 100 microns. A fixed quantity of each of these crushed coal samples were taken in a Petri dish as shown in Fig.3.1 and heated uniformly using a microwave oven at 850W power level for a duration of 40s which was found to be the optimum time for compositional analysis of coal.



**Fig. 3.1 Coal samples: 1. Sample C141 2. Sample D37**

### 3.2 IR imaging of coal

At the end of exposure time, the Petri dish with ore specimens is removed away from the heat source and placed under the IR camera. Infrared Thermography system (make FLIR Agema 550) with  $0.1^{\circ}\text{C}$  resolution is configured to capture the thermal image of the heat radiated by iron ore specimens placed at fixed distance from the IR camera as shown in Fig. 3.2.



**Fig. 3.2 Schematic for IR Thermography of coal samples**

The specification of IR camera used for infrared imaging are:-

- ✓ **Make & Model** : FLIR Agema, Thermovision 550
- ✓ **Temperature range** :  $-20^{\circ}\text{C}$  to  $500^{\circ}\text{C}$
- ✓ **Sensitivity** : Less than  $0.1^{\circ}\text{C}$
- ✓ **Pixels** : 320 X 240
- ✓ **Spectral Range** :  $3.6\text{-}5\text{ }\mu\text{m}$

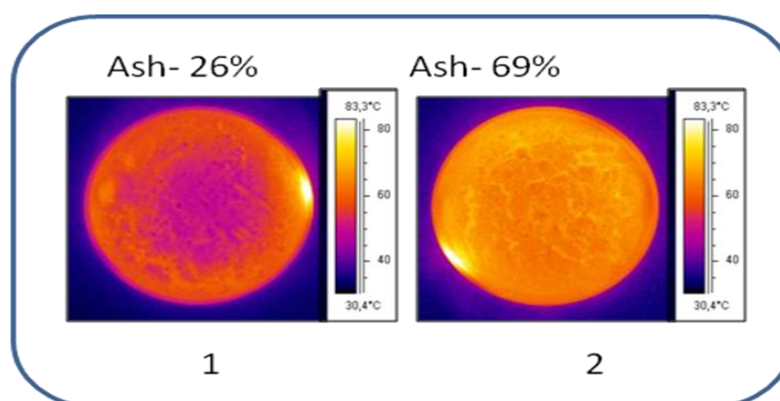


The Fig. 3.3 shows the IR camera and set up for thermal imaging whose real time image is displayed on the television screen.



**Fig. 3.3 IR camera and experimental set-up.**

IR Thermography System is configured to capture the thermal image of the heat radiated by the coal samples placed at fixed distance from the camera as shown in Fig. 3.4. To take into account the change in moisture content in the coal samples due to heat, each of the samples are weighed before after the experiments.

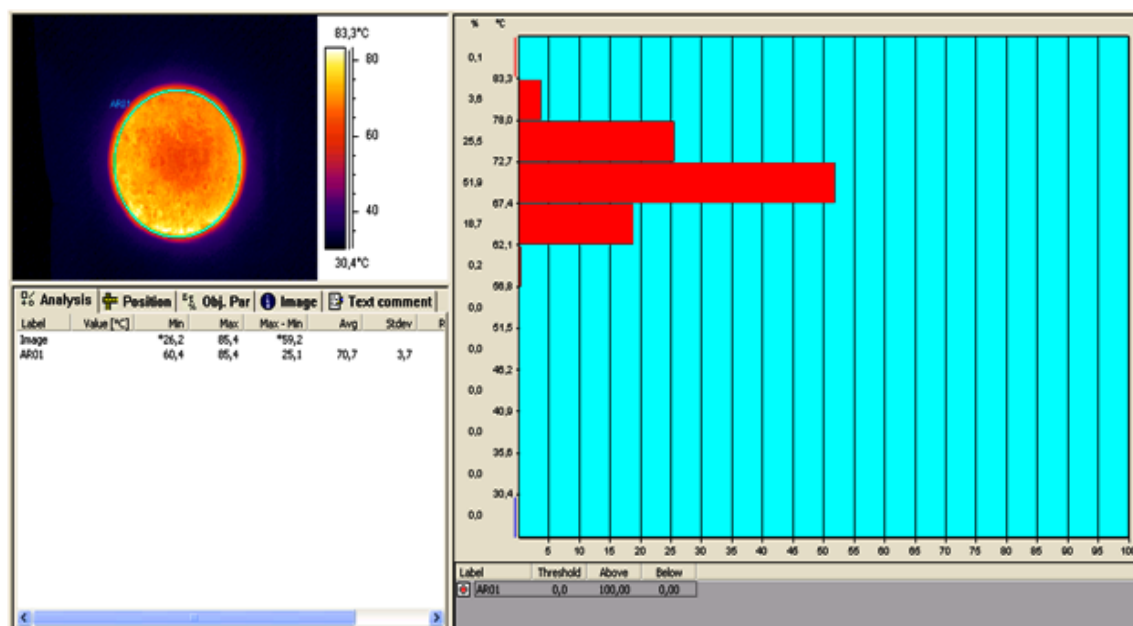


**Fig. 3.4 Thermal image of coal samples: 1. Sample C141 2. Sample D37**

### 3.3 Image analysis

The radiant heat from the coal sample is captured as a thermal image and the temperature profile of samples analyzed using IR Thermography software ThermaCAM Researcher Pro 2.8 as shown in Fig 3.5. The area of interest in the thermal image is selected. The average peak temperature for each of the fixed quantity of heated ore samples is determined from the histogram plot of temperature variation

in the selected area. The average temperature of each portion is high or low depending upon the ash and moisture content present in them.



**Fig. 3.5 IR image analysis of a microwave heated coal sample**

### 3.4 Calibration curves for Ash and Moisture Determination

The average temperature of each sample has been obtained from IR imaging and the ash and moisture content in these samples is obtained through chemical analysis. To study the effect of ash and moisture on thermal behaviour of heated coal, the curve between average peak temperature and ash and moisture content in coal samples has been plotted. The obtained plot can be used as a reference to estimate the ash and moisture content in coal samples without the help of time-consuming and cumbersome chemical analysis approach. The steps followed for the prediction of ash / moisture present in coal samples is shown in Fig. 3.6 in the form of a flowchart.

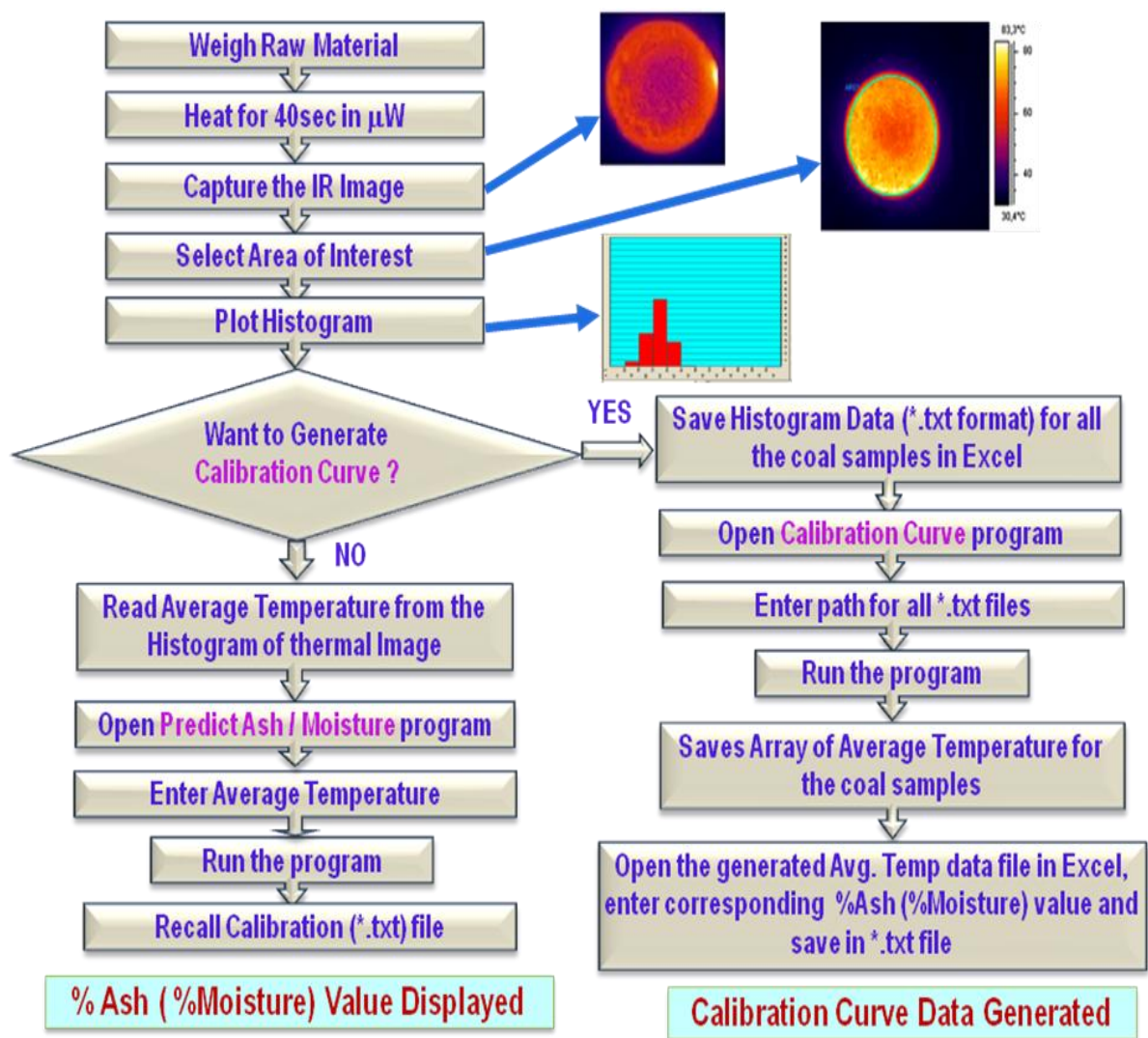


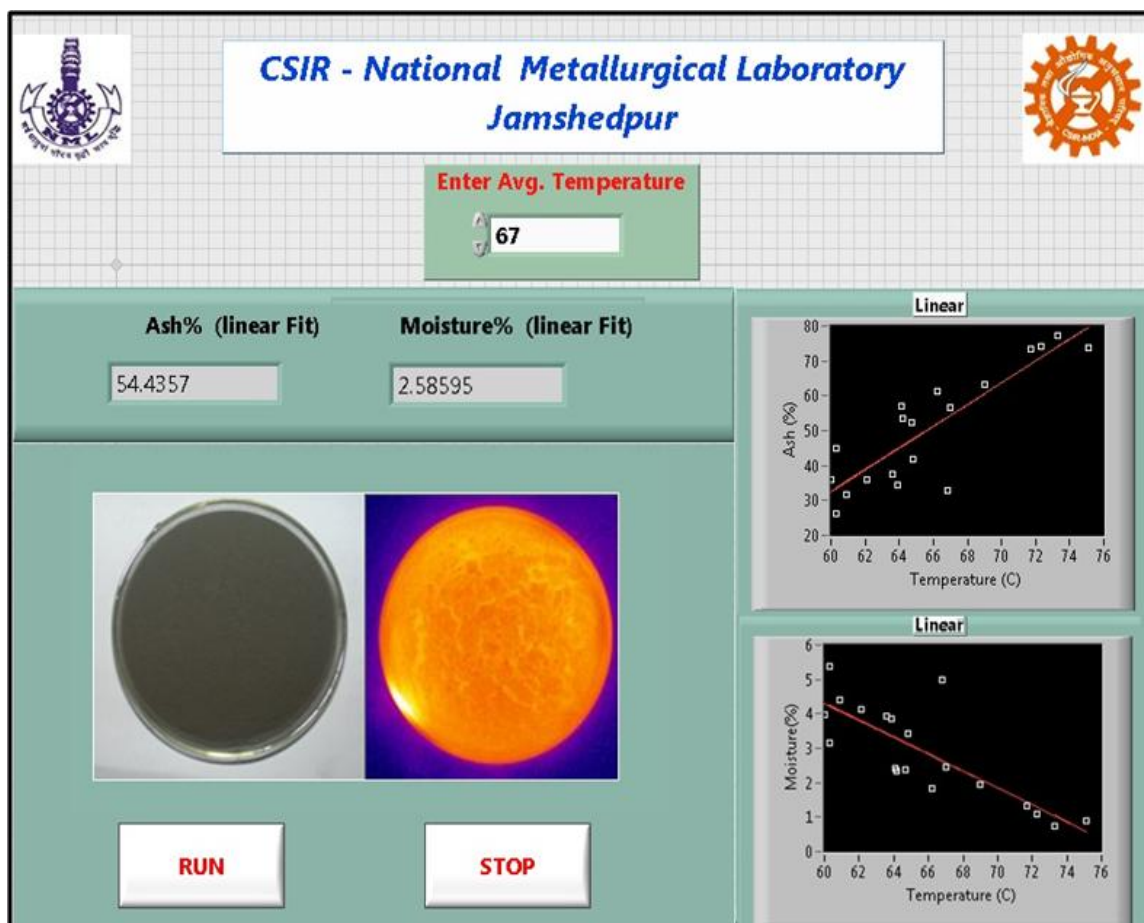
Fig. 3.6 Steps for ash and moisture prediction

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 LabVIEW program to predict ash and moisture content

A program has been developed in LabVIEW for prediction of ash and moisture content in coal, the front panel for which has been shown in Fig. 4.1. The program is capable of displaying the ash and moisture content coal samples on entering its average temperature, obtained from the captured IR image of the sample.



**Fig.4.1 Software to predict ash and moisture**

With the help of this software the ash and moisture content in coal can be predicted in a much more faster and easier way as compared to the chemical analysis.

## **4.2 IR THERMOGRAPHY OF COAL SAMPLES**

### **4.2.1 Coal samples from Sambaleswari block**

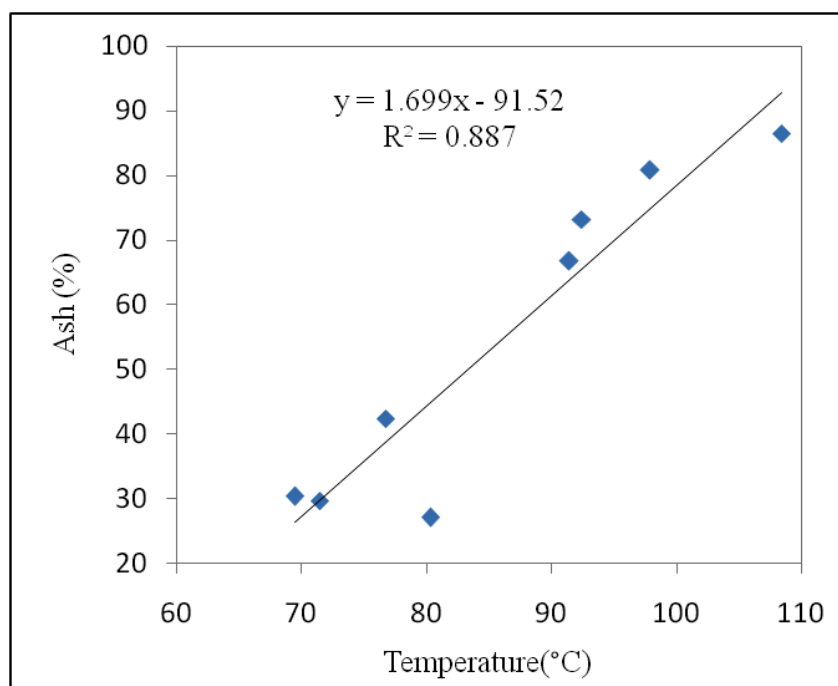
The average temperature of the samples obtained from IR imaging of coal and the ash and moisture content in these samples from proximate analysis for Set I and Set II samples are given in Table 4.1 and Table 4.2 respectively. By entering the the average temperature obtained from IR imaging, the ash and moisture content in coal is predicted using the respective calibration curve with the help of software shown in

Fig. 4.2. The error between the predicted and actual value is also estimated and given in the Table 4.1.

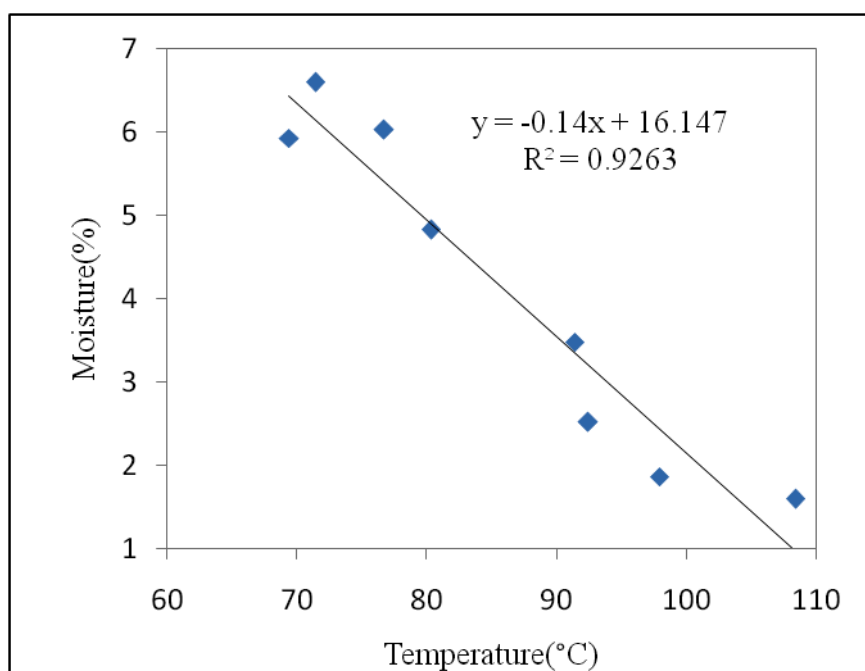
**Table 4.1 Analysis results for Set I samples**

Sample ID	Average Temperature (°C)	Ash (%)			Moisture (%)		
		Chemical analysis	Predicted	Error	Chemical analysis	Predicted	Error
C77	71.4	29.73	29.84	-0.37	6.6	6.15	6.8
C56	71.4	30.38	29.84	1.78	5.93	6.15	-3.7
D28	80.3	27.19	44.97	-3.23	4.83	4.9	-1.44
C22	76.7	42.49	38.85	-5.84	5.43	5.41	0.36
D10	91.4	66.75	63.84	4.35	3.48	3.35	3.73
D1	92.4	67.24	65.54	2.52	3.42	3.21	6.14
D94	97.9	80.81	74.88	7.33	2.47	2.44	1.21
D90	108.4	86.52	92.73	-7.17	1.05	0.97	7.61

The variation in average temperature of heated coal samples with the corresponding ash and moisture content for Set I samples have been plotted in Fig. 4.2. and 4.3 respectively.



**Fig.4.2 Ash vs Temperature plot for Set I**



**Fig. 4.3 Moisture vs Temperature content plot Set I**

A correlation factor ( $R^2$ ) of 0.887 and 0.9263 has been obtained for the calibration curves which indicate that IR imaging can be used for the determination of ash and moisture content in the coal. It can be clearly observed from the plot that the ash and moisture present in the coal samples has a direct influence on the change in average temperature of the heated samples. If the ash in the coal samples is more, the average temperature of the heated coal samples is found to be higher while it is reverse with coal samples having high moisture. The obtained calibration curves can be used to estimate the ash and moisture content in coal samples without the help of any chemical analysis.

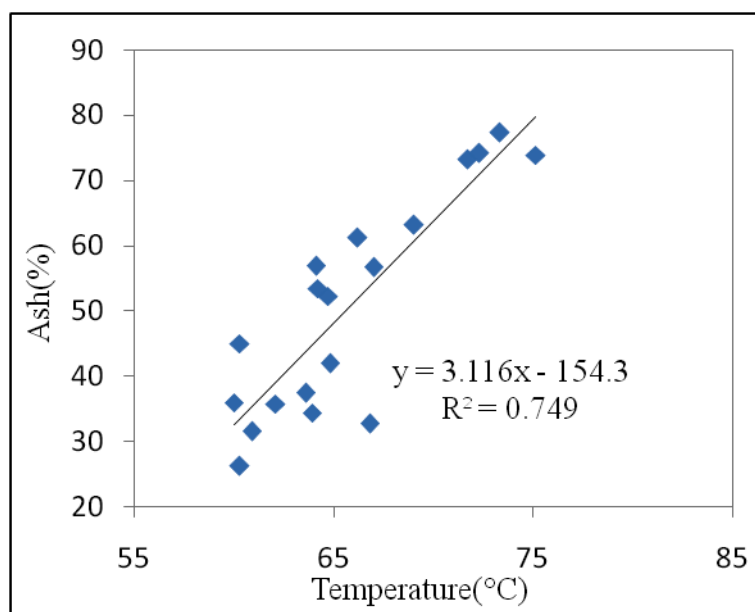
The average temperature of the samples obtained from IR imaging of coal and the ash and moisture content in these samples from chemical analysis of Set II samples are given in Table 4.2. By entering the the average temperature obtained from IR imaging, the ash and moisture content in coal is predicted from the calibration curve. The predicted values of ash and moisture and the percentage error are given in the Table 4.2.

**Table 4.2 Analysis results for Set II coal samples**

Sample ID	Average Temperature (°C)	Ash (%)			Moisture (%)		
		Chemical analysis	Predicted	Error	Chemical analysis	Predicted	Error

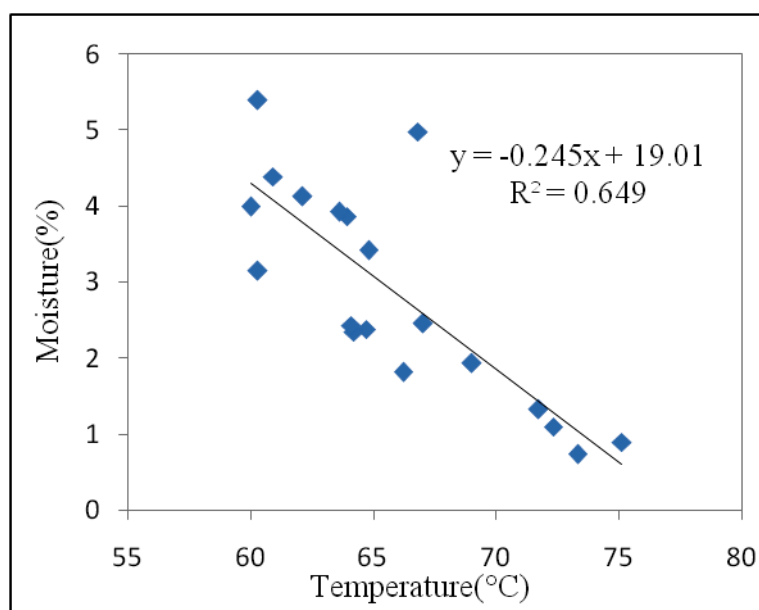
C124	60.3	31.29	33.55	-7.22	4.39	4.23	3.64
C41	66.8	50.76	53.81	-6.01	2.88	2.64	8.33
C143	60.9	33.56	35.42	-5.54	4.39	4.08	7.06
C142	62.1	35.78	39.16	-9.45	4.13	3.79	8.23
C141	63.9	42.4	44.77	-5.59	3.47	3.35	3.46
C122	60	35.98	32.61	9.37	3.99	4.3	-7.77
C5	63.6	40.41	43.84	-8.49	3.63	3.42	5.79
C3	64.8	45.93	47.58	-3.59	3.42	3.13	8.48
C11	60.3	35.03	33.55	4.22	4.15	4.23	-1.93
D47	64.7	50.27	47.26	5.99	3.28	3.15	3.96
D49	64.2	48.49	45.71	5.73	3.35	3.27	2.39
D16	64.1	46.92	45.4	3.24	3.43	3.3	3.79
D14	67	56.69	54.43	3.99	2.46	2.58	-4.88
D5	69	63.2	60.67	4.00	1.94	2.09	-7.73
D32	66.2	52.22	51.94	0.54	2.82	2.78	1.42
D44	71.7	73.19	69.08	5.62	1.33	1.43	-7.52
D35	72.3	74.23	70.95	4.42	1.18	1.28	-8.47
D37	75.1	73.88	79.68	-7.85	0.85	0.79	7.06
D39	73.3	77.3	74.07	4.18	0.97	1.04	-7.22

The variation in average temperature of heated coal samples with the corresponding ash and moisture content for the second set of samples have been plotted in Fig. 4.4 and 4.5 respectively.





**Fig.4.4 Ash content vs Temperature plot for Set II samples**



**Fig. 4.5 Moisture vs Temperature content plot for Set II samples**

A correlation factor ( $R^2$ ) of 0.749 and 0.649 has been obtained for the calibration curves. It can be clearly observed from the graph that the ash and moisture content is influencing the thermal behavior of the coal samples. The ash in the coal samples leads to increase in temperature and the moisture causes the temperature to fall when the coal samples are heated. Therefore the average temperature of the heated coal samples is directly proportional to the ash and inversely proportional to the moisture present in the samples. The obtained calibration curves can be used as a reference to estimate the ash and moisture content in coal samples without the help of any chemical analysis.

#### **4.2.2 Coal samples from Talcher block**

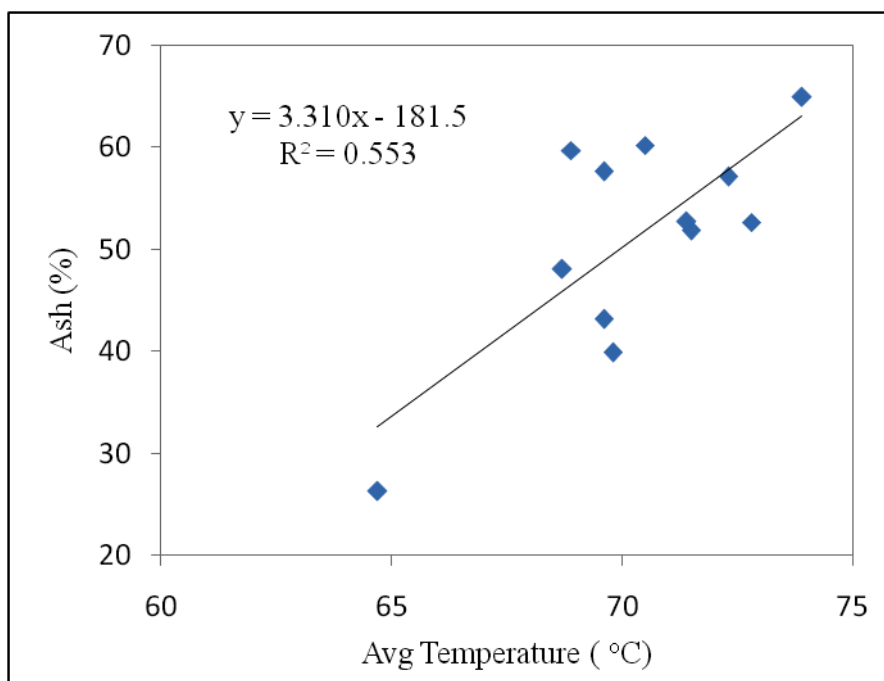
Two sets of coal samples from Talcher block have been collected from the Coal Characterization Centre in CSIR-NML. As mentioned in Section 3.1, these samples were heated in a microwave for duration of 40s. The average temperature of the heated coal samples obtained by IR imaging is given for Set III and Set IV samples in Tables 4.3 and 4.4 respectively. The corresponding ash and moisture content in these samples from chemical analysis are also given in Table 4.3 and Table 4.4. The ash and moisture content in coal samples is predicted from the relevant calibration curve and the percentage error is also estimated which are given in the respective tables.

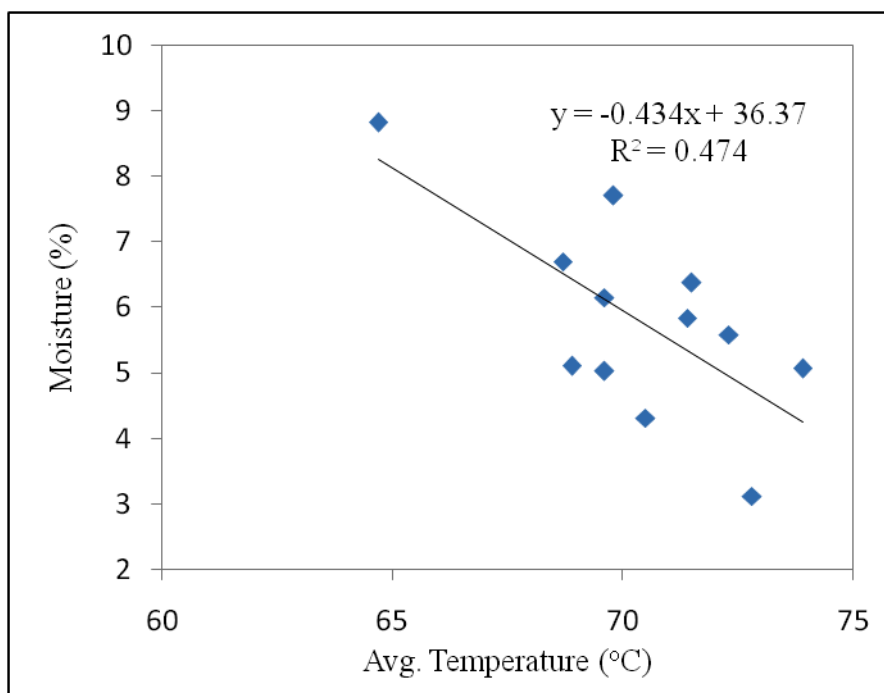


**Table 4.3 Analysis results for Set III coal samples**

Sample ID	Average Temperature (°C)	Ash (%)			Moisture (%)		
		Chemical analysis	Predicted	Error	Chemical analysis	Predicted	Error
C113	69.6	57.63	48.86	15.22	6.32	6.12	3.16
C117	68.9	49.73	46.54	6.41	6.1	6.42	-5.25
D31	70.5	60.24	51.84	13.94	5.31	5.73	-7.91
C52	69.6	45.2	48.86	-8.10	6.15	6.12	0.49
C40	71.5	51.88	55.15	-6.30	5.88	5.3	9.86
C170	64.7	36.3	32.64	10.08	8.82	8.25	6.46
C167	68.7	48.11	45.88	4.64	6.7	6.5	2.99
C49	69.8	47.87	49.52	-3.45	6.7	6.04	9.85
C38	71.4	52.75	54.82	-3.92	5.84	5.34	8.56
D26	72.3	57.11	57.8	-1.21	5.58	4.95	11.29
D49	73.9	64.98	63.1	2.89	5.16	4.25	17.64
D130	72.8	57.65	59.45	-3.12	4.22	4.73	-12.09

The variation in average temperature of heated coal samples with the corresponding ash and moisture content for Set III samples have been plotted in Fig. 4.6. and 4.7 respectively.

**Fig.4.6 Ash vs Temperature plot for Set III samples**



**Fig. 4.7 Moisture vs Temperature content plot for Set III samples**

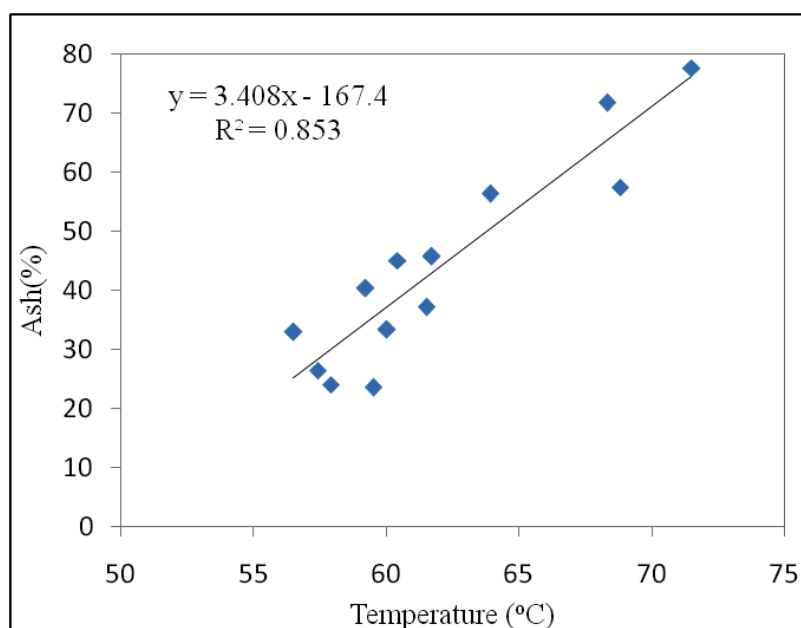
A correlation factor ( $R^2$ ) of 0.553 and 0.474 has been obtained for the calibration curves which are less compared to the other set of samples and it can be attributed to the variation in ash contents in coal samples of different mines. The different metal oxides present in the ash can influence the thermal behavior of the coal samples.

**Table 4.4 Analysis results for Set IV of coal samples**

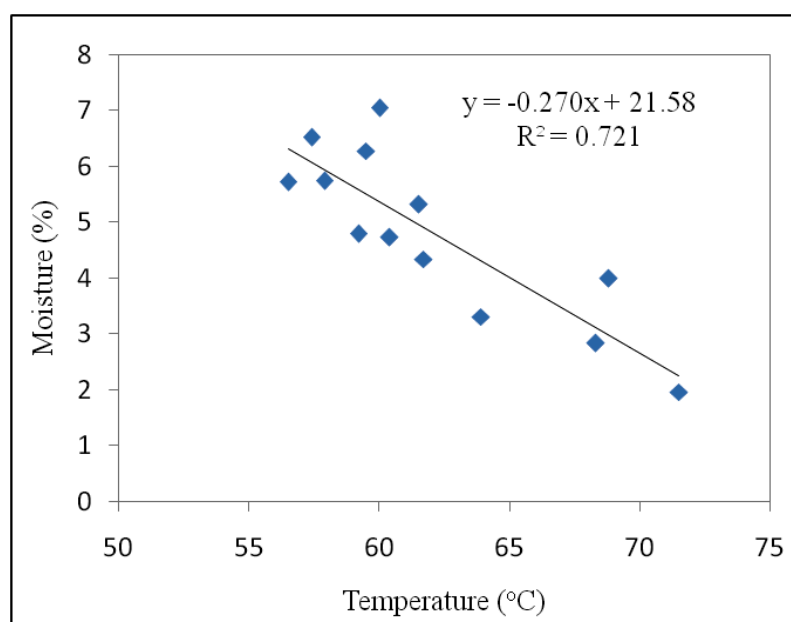
Sample ID	Average Temperature (°C)	Ash (%)			Moisture (%)		
		Chemical analysis	Predicted	Error	Chemical analysis	Predicted	Error
D320	68.8	65.57	67.1	-2.33	2.79	2.99	-7.17
C274	57.9	27.16	29.95	-10.27	5.74	5.93	-3.31
D254	68.3	71.89	65.4	9.03	2.84	3.12	-9.86
D252	71.5	77.71	76.31	1.80	1.96	2.16	-10.20
C235	61.7	45.83	42.9	6.39	4.64	4.9	-5.60
C233	59.2	38.48	34.38	10.65	5.79	5.58	3.63
C231	56.5	23.02	25.17	-9.34	5.72	6.31	-10.31
D266	60.4	40.1	38.47	4.06	4.93	5.26	-6.69
C230	57.4	26.55	28.24	-6.37	6.52	6.07	6.90
D262	63.9	54.47	50.4	7.47	4.11	4.31	-4.87

C229	61.5	37.23	42.22	-13.40	5.12	4.96	3.13
C228	60	33.39	37.1	-11.11	6.05	5.37	11.24
C227	59.5	33.66	35.4	-5.17	6.28	5.5	12.42

The variation in average temperature of heated coal samples with the corresponding ash and moisture content for Set IV samples have been plotted in Fig. 4.8. and 4.9 respectively.



**Fig. 4.8 Ash vs Temperature content plot for Set IV samples**



**Fig. 4.9 Moisture vs Temperature content plot for Set IV samples**

A correlation factor ( $R^2$ ) of 0.721 and 0.853 has been obtained for the calibration curves. Just as in case of Set I and Set II samples from Sambaleswari block and Set IV samples from Talcher block, it is observed from the plots in Fig 4.8 and 4.9, that variation in ash and moisture content in the coal samples results in change of average temperature the heated coal samples. The obtained calibration curves can be used as a reference to estimate the ash and moisture content in coal samples without the help of any chemical analysis.

## **CHAPTER 5**

### **SUMMARY OF WORK**

#### **5.1. INFERENCES**

1. A fast, low cost and non-invasive technique for compositional analysis of coal based on IR Thermography of microwave heated coal samples has been formulated.
2. Ash and moisture content in coal samples can be determined from the calibration curve using the software developed in LabVIEW.
3. IR Thermography-based technique for compositional analysis can be considered as an alternative for the more prevalent proximate analysis method which is time-consuming and cumbersome.
4. As the constituents of ash vary from mines to mines, a separate calibration curve needs to be obtained for each of the mines.
5. The IR Thermography can be successfully implemented for compositional analysis of the coal and opens a new horizon in non-destructive testing applications for mineral gradation.

#### **5.2. FUTURE WORK**

1. Implementation of the coal compositional analysis technique in the beneficiation process in operating mines with automatic chemical report generation facility.
2. Further research in this area would help in achieving an IR thermography based efficiently operating mine-scale coal sorting system.

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